Univ. of
Toronto
Library

BINDING LIST NOV 1 1923

Digitized by the Internet Archive
in 2011 with funding from
University of Toronto

http://www.archive.org/details/technologicpaper109unit

Gov. Doc
U.S.
S
U.S. Standards, Nat. Bur. of
T
1
U55
no.109-120
LIBRARY
FEB 5 1971

DEPARTMENT OF COMMERCE
BUREAU OF STANDARDS
S. W. STRATTON, Director

185807
29.11.23

TECHNOLOGIC PAPERS OF THE BUREAU OF STANDARDS NO. 109 ~ 120.

Issued March 15, 1919.

CONSERVATION OF TIN IN BEARING METALS, BRONZES, AND SOLDERS

By G. K. Burgess, Physicist, and R. W. Woodward, Assistant Physicist

CONTENTS

		Page
I.	Introduction	1
II.	Bearing metals	1
III.	Standard grades of Babbitt metal	4
IV.	Structural bronzes	5
V.	Solders	7
VI.	Cadmium as tin substitute	8

I. INTRODUCTION

From a metallurgical standpoint there are several ways in which a reduction of the tin consumed in commercial nonferrous and white-metal alloys can be effected: First, a reduction of the tin content of the alloy; second, substitution of part or all of the tin content by some other metal; and third, a substitution of a different type of alloy, which in some cases also involves a change in mechanical design. The Bureau of Standards has been studying these methods of conservation for tin alloys, particularly in regard to babbitts and bearing metals, bronzes, and solders.

Much of the information secured by the Bureau was obtained from answers to questionnaires sent to manufacturers and users of these materials, such that, in general, any suggestions or recommendations made can be considered as being practical and as having already been thoroughly tried.

II. BEARING METALS

There is no question but that the tin content of nearly all bearing metals can be reduced to some extent, and in some cases actually eliminated, without prejudice to the service rendered.

1918-1919

So the problem is to determine what needs are the most exacting or when a breakdown would cause the greatest damage and confine the use of high tin babbitts to these uses only. Thus the main bearings of airplane and military automobile engines, turbine shafts, etc., will probably have to continue to use high tin babbitt with a tin content of from 84 to 91 per cent. A babbitt metal such as S. A. E. specification No. 24 containing 84 per cent of tin, 9 of antimony, and 7 of copper appears to be as satisfactory in service as genuine babbitt—89 per cent of tin, 7½ of antimony, and 3½ of copper—or that specified by the International Aircraft Standards Board—91 per cent of tin, 4½ of antimony, and 4½ of copper; but it should be pointed out that the latter two compositions are more fluid in the molten condition than the first named and consequently the lining can be made in a thinner shell with these babbitts, and the total amount of tin consumed may therefore be less than if the S. A. E. No. 24 were used. However, if the design of the bearing is not altered to admit of the thinner shell, the lower composition babbitt should be used in general.

The following compositions are also recommended for use where a high grade of lining is required and where a genuine babbitt is often now used:

Ingredients	No. 1	No. 2	No. 3	No. 4	No. 5	No. 6
	Per cent	Per cent	Per cent	Per cent	Per cent	Per cent
Tin	65	62	8	5	10	21.3
Antimony	.....	.....	8	7	12.5	.....
Copper	3- 6	4	4	2	.5	3.0
Zinc	28-30	33	.....	76	.....	63.3
Aluminum	.....	1	.....	.....	.....	.....
Lead	.....	.....	80	10	77	12

Nos. 3 and 4 have been found to do the service required of tin base linings in machine-tool bearings. No. 5 can be used on similar bearings where a greater strain is met. No. 6 is in use in Germany as a "best" babbitt to conserve both tin and copper.

For linings on railroad-truck journals two compositions are in general use, one composed of 85 per cent of lead, 10 of antimony, 5 of tin, and the other of 87 per cent of lead and 13 of antimony. The latter is restricted by some roads to freight service and the former used on passenger equipment. Many roads, however, use the 87 per cent of lead and 13 of antimony on both classes of service, and it seems that its use might be made more universal.

Another type of lining metal which deserves serious consideration is one composed almost entirely of lead, with small additions

of alkali or alkali earth metal. Certain of these have been given service tests at the Bureau of Standards and in many respects were found equal to or superior to genuine babbitt.

Following is a summary of the tests on the metal and similar tests on genuine babbitt of composition 89 per cent of tin, 7½ of antimony, and 3½ of copper.

GENUINE BABBITT

Load, pounds per square inch	Revolutions per minute	Total number of revolutions	Final temperature		Rise in temperature		Friction	Loss in weight	Remarks
			°C	°F	°C	°F	Pounds	Grams	
100	694	12 230	89	192	53	95	22	0.023	
200	706	16 510	102	216	58	104	29	.021	
300	682	15 150	125	257	100	180	38	.013	Belt slipping
400	603	6 600	139	282	94	169	79	.054	Bearing seized and smoking

ULCO HARD METAL

100	710	13 160	56	133	23	41	13	0.013	
200	715	18 870	69	156	33	59	18	.021	
300	719	18 830	80	176	42	76	27	.013	
400	711	17 310	81	178	43	77	23	.022	
500	723	17 660	79	174	43	77	25	.014	
600	692	14 960	84	183	45	81	24	.021	
700	648	24 520	62	144	38	68	24	.020	
800	365	12 870	53	127	20	36	23	.010	
900	408	22 300	59	138	22	40	24	.015	
1000	405	23 200	66	151	36	65	22	.014	Bearing still in good condition

Large quantities of phosphor bronze of the compositions 80 per cent copper, 10 lead, and 10 tin, and deoxidized with phosphorus, are used in unlined bearings at fairly high speeds and pressures. The following compositions have been suggested as substitutes for this composition, although it is our opinion that trouble will sometimes be experienced with Nos. 8, 9, and 10 because of the high lead content, and they have about the same tin content as the others in the list:

Ingredients	No. 7	No. 8	No. 9	No. 10	No. 11	No. 12
Copper	81	79	74	64	Remainder	Remainder
Tin	7	5	5	5	8	5
Lead	9	15	20	25	15	17.5
Zinc	3	.....	.....	.....	1.5–3	5
Antimony	.....	.....	.....	5	.....	.....
Phosphor copper	.....	1	1	1	.....	.....

III. STANDARD GRADES OF BABBITT METAL

At a meeting called by the conservation division of the War Industries Board on April 15, 1918, which was attended by manufacturers and users of bearing metals, the Bureau of Standards was requested, after conference with technical representatives of the large manufacturers and users, to determine if four classes of babbitt metal could be adopted ranging in tin content as follows:

A (genuine babbitt)	89 per cent tin.
B	40 to 50 per cent tin.
C	4 to 6½ per cent tin.
D	No tin.

The Bureau has gone over the situation with several of the representatives as noted above, and the general opinion seems to be that it is impossible to limit some of the classes to a single composition, because of the fact that several compositions of nearly the same tin content are in general use for different purposes. Thus, in the table below, No. A1 is used in aircraft engines, No. A3 is used for automobile engines, and No. A4 is found in bearings of electrical machinery. It was thought, however, that Class B can be entirely dispensed with, as these intermediate tin content-bearing metals are in no way as satisfactory as either a high lead or high tin base babbitt. In all cases where Class B could be used, Class C or Class D will be found to serve the purpose equally as well. There are, however, some grades of babbitt containing about 65 per cent of tin which do not fall into either Class A or Class B, but which are often claimed by the manufacturers to equal the high tin babbitt in performance. If these claims can be substantiated, this babbitt should be considered as falling into the category of Class A and as being a substitute for alloys in that class.

It should not be presumed that because high tin babbitt of Class A are included that the Bureau recommends the continuance of its use for many bearings in which it is now used. The lowest possible tin-content alloy should always be used, and the Bureau believes that it might be advisable to have some central body issue licenses for the use of babbitt in Class A in order to insure that no Class A babbitt is being used where others are satisfactory.

Alloy D2 has been included in Class D because this comprises babbitt metals containing no tin. It should be noted, though, that this alloy will be found satisfactory in many installations

where Class A has hitherto been used, and its inclusion in Class D should not give the impression that it is a low-grade babbitt.

The American Society for Testing Materials has drawn up specifications for 12 compositions of babbitt metals (B23–18T) which, however, do not take into consideration the factor of tin conservation, but are formed for use in peace times. The present recommendations are for use in the existing situation where the saving of all tin possible is of prime importance.

The recommended compositions for the various classes are given in the table following. These compositions have been selected so as to include existing specifications and usage of babbitt metals as far as possible. Much information has been secured from the questionnaire sent out by the Bureau and from replies to the general letter issued by the War Industries Board on May 29, 1918.

Class	No.	Tin	Antimony	Lead	Copper	Iron [a]	Arsenic [a]
		Per cent	Per cent	Per cent	Per cent		
A	A1	91	4½	[a] 1.00	4½	0.03	0.10
	A2	89	7½	[a] 1.00	3½	.08	.10
	A3	84	9	[a] 1.00	7	.08	.10
	A4	83	8½	1.00	8½	.08	.10
C	C1	5	10	85	[a] 50	.....	.20
D	D1	.....	13	87	[a] 50	.....	.25
	D2	Lead about 98 per cent, balance alkali and/or alkali earth					

[a] Maximum.

More than traces of impurities other than those listed above will not be allowed; the following variations above or below the specified amount will be permissible for the desired elements:

Per cent of elements specified	Permissible variations over or under the specified value units of per cent
Not over 5 per cent	0.50
5 to 10 per cent, inclusive	.75
Over 10 per cent	1.00

IV. STRUCTURAL BRONZES

Passing now to the bronzes other than those used for bearing purposes, we find that "Government bronze" (Navy Specification 46M6a, gun metal) of 88 per cent copper, 10 tin, and 2 zinc, is used in large quantities and can also be modified to admit of a saving

of tin without impairment of the physical properties sought. Experiments by the Bureau of Standards and others have shown that a composition of 88 per cent copper, 8 tin, and 4 zinc, is equal to or superior to the ordinary Government bronze. Aluminum bronze of composition 90 per cent aluminum and 10 per cent copper, for example, can also be substituted for many uses of Government bronze, as can also manganese bronze and naval brass. There have also been introduced several aluminum bronzes containing small amounts of iron, which can be either cast or wrought and which are now being used by several former users of Government bronze.

Some manufacturers have raised an objection to using aluminum bronze because the scrap accumulating from this alloy, if it gets mixed with other metals, particularly valve metals, would have a deleterious effect upon them. This is simply a problem in works management of properly sorting and routing scrap. We know of several large manufacturers who make aluminum-bronze castings in the proximity of steam metal castings and who by taking the proper precautions have encountered no difficulties.

The following table gives some of the properties of the above-mentioned alloys:

Alloy	Tensile strength	Elastic limit	Elongation in 2 inches	Authority
	Lbs. in.^{2}	Lbs. in.^{2}	Per cent	
Government bronze, 88 Cu, 10 Sn, 2 Zn.	38 860	12 250	25.2	Average of 30 tensile specimens poured in 5 different foundries. Tensile tests made at the Bureau of Standards.
88 Cu, 8 Sn, 4 Zn	39 220	11 000	30.6	Same as above, except that it represents only 26 specimens.
Aluminum bronze	71 000	25 000	21	Corse & Comstock, Trans. Soc. Autom. Eng., 1916, Part II, pp. 272-273.
Manganese bronze, U. S. N., Mn.	70 000	35 000	30	Navy Department specification 46B15.
Naval brass	[a] 61 000	25 000	40	Navy Department specification 46B6b.
	[b] 60 000	27 000	35	
Aluminum bronze with iron Sillman bronze:				
Wrought alloy [c]	84 400	14 000	11.5	Bureau of Standards test.
Cast alloy	78 850	11 500	14.5	Do.

[a] Over 1 inch.
[b] Below ½ inch.
[c] Chemical compositions. Cu, 86.4 per cent; Al, 9.7 per cent; Fe, 3.9 per cent.

At the suggestion of the Imperial Electric Co. of Akron, Ohio, the Bureau also made tests on Government bronze in which one-half of the tin was replaced by an equal amount of nickel. The

averages of several tests on these alloys are shown in the table below.

Composition	Ultimate strength	Yield point	Elongation in 2 inches	Reduction in area	Modulus of elasticity
	Lbs. in.^{2}	Lbs. in.^{2}	Per cent	Per cent	Lbs. in.^{2}
Cu 88, Sn 5, Ni 5, Zn 2	40 500	13 050	31 8	28.0	17 300 000
Cu 89, Sn 4, Ni 4, Zn 3	36 875	11 500	31 2	31 2	14 900 000

It will be observed that the above values are exceedingly good for this class of material and that either of the above alloys can be used as a means of conserving tin.

Many small machine-parts castings are made of bronze, which can just as readily, or even sometimes better, be manufactured of a brass composition—i.e., copper-zinc alloys. Tin is also looked upon in many brass foundries as a "cure all" for poor castings and is often added to the mixture when trouble is encountered. This practice is not only questionable and should not obtain in brass foundries, but the cause of the poor castings should be determined and the proper remedies applied in a regular manner.

V. SOLDERS

The composition of solders will vary with the use for which the solder is intended. Formerly, every mechanic believed that nothing was as good as "half-and-half," or 50 per cent tin and 50 per cent lead solder. There are very few cases where a 50–50 solder is necessary in the present emergency; its use should be eliminated. No solder over 45–55 should be used for hand soldering with the iron and in the majority of cases 40–60 will serve the purpose. Most plumbers use 40–60 for making wiped joints, whereas 37½–62½ is for all such purposes just as satisfactory. Up to 1½ per cent of the tin in a wiping solder can be replaced by antimony, although this element is objectionable if present in solder for other purposes.

In the manufacture of automobile or airplane radiators very little solder need be used of greater tin content than 40 per cent, and in many cases 35–65 or lower is being used with success. For the canning industry, both in the manufacture of the can and in its sealing, 37½–62½ solder can be used with satisfactory results. Articles which are tinned previous to soldering can be "tinned" in a bath composed of the eutectic of tin and lead—63 per cent tin and 37 per cent lead. This composition will be found to be fluid and will not segregate, as will certain other tin-lead baths.

VI. CADMIUM AS TIN SUBSTITUTE[1]

Cadmium appears as a promising substitute for part of the tin in solders. The Bureau has been developing such a solder, and laboratory test, together with manufacturing experience, so far point to a composition of 80 per cent lead, 10 per cent tin, and 10 per cent cadmium as being practical for many of the purposes for which solder is required. This solder has been tried in the manufacture of tin cans, on roofing materials, and for electrical joints, with encouraging results in all cases. Before using it for food containers, however, it will be necessary to ascertain its toxic properties under various conditions. A test has also been made of it in the manufacture of automobile radiators with most satisfactory results.

The tensile strength of the cadmium solder is about the same as that of 40–60 solder, but the ductility is approximately twice that of the ordinary solders. The point of complete liquidation is only slightly higher than that of the ordinary composition of solders, while the range of solidification is considerably greater. The following table gives some of the provisional data on these solders as compared to the tin-lead solders. The tensile properties given are the average of four determinations and were made on a Scott testing machine, the rate of separation being about 12 inches per minute

Physical Properties of Cadmium-Lead and Tin-Lead Solders

	Initial solidification	Secondary solidification	Final solidification	Specific gravity	Equivalent volume to 1 volume 50–50 solder	Tensile strength	Elongation in 2 inches
	°C	°C	°C			Lbs./in.²	Per cent
50 per cent tin and 50 per cent lead	[a] 210	[a] 181	[a] 149	8.81	1.00	5698	20.3
40 per cent tin and 60 per cent lead	[a] 238	[a] 181	[a] 149	9.47	1.07	5820	26.0
37½ per cent tin and 62½ per cent lead	[a] 245	[a] 181	[a] 149	9.54	1.08	5383	28.8
90 per cent lead and 10 per cent cadmium	[b] 267	.....	[b] 249	11.09	1.26	5000	37.5
80 per cent lead, 10 per cent cadmium, and 10 per cent tin	254	183	143	10.35	1.17	5727	52.3
85 per cent lead, 10 per cent cadmium, and 5 per cent tin	257	202	141	10.67	1.21	(c)	(c)
75 per cent lead, 10 per cent cadmium, and 15 per cent tin	(c)	(c)	(c)	10.26	1.16	5880	41.7

[a] Rosenhain and Tucker, Phil. Trans. A. 209, p. 89; 1918.
[b] Kapp Tucker Ann. 6, p. 264; 1921.
[c] Not determined.

For most of the experimental data on cadmium solders the authors are indebted to L. J. Gurevich of the metallurgical staff of the Bureau.

Because of the preponderance of lead in the cadmium solder, the price of it is very reasonable; with the present market prices of the metals involved it is thought that the 80–10–10 solder can be sold at a profit of 35 cents a pound. It is also thought that plenty of cadmium can be produced as soon as the market for it is created, as there are undoubtedly available American sources of cadmium which are not at present exploited.[2]

In meetings with manufacturers of materials containing tin it is always brought out that the Government is the worst offender, and that many Government specifications often call for a lavish use of tin which is sometimes detrimental to the quality of the material manufactured. We believe, however, that many of these specifications are being revised in order to conserve tin, but that there is still room for further improvement. As a means of conserving the use of tin by the Government, we would suggest the advisability of creating a joint committee of technical representatives of the various departments to pass upon or revise all Government specifications containing tin. Such a committee could be in close cooperation with the manufacturers and would form a better means for the manufacturers to criticize the tin content of Government specifications than now exists in any of the departments.

WASHINGTON, October 21, 1918.

[2] See C. E. Siebenthal, Cadmium in 1917, Mineral Resources of United States, Pt. I, p. 49–53; 1917.

ADDITIONAL COPIES
OF THIS PUBLICATION MAY BE PROCURED FROM
THE SUPERINTENDENT OF DOCUMENTS
GOVERNMENT PRINTING OFFICE
WASHINGTON, D. C.
AT
5 CENTS PER COPY

DEPARTMENT OF COMMERCE

TECHNOLOGIC PAPERS

OF THE

BUREAU OF STANDARDS

S. W. STRATTON, DIRECTOR

No. 110

INFLUENCE OF QUALITY OF GAS AND OTHER FACTORS ON THE EFFICIENCY OF GAS-MANTLE LAMPS

BY

R. S. McBRIDE, Engineer Chemist
W. A. DUNKLEY, Associate Gas Engineer
E. C. CRITTENDEN, Associate Physicist
A. H. TAYLOR, Assistant Physicist

Bureau of Standards

ISSUED OCTOBER 25, 1918

PRICE, 15 CENTS

Sold only by the Superintendent of Documents, Government Printing Office, Washington, D. C.

WASHINGTON
GOVERNMENT PRINTING OFFICE
1918

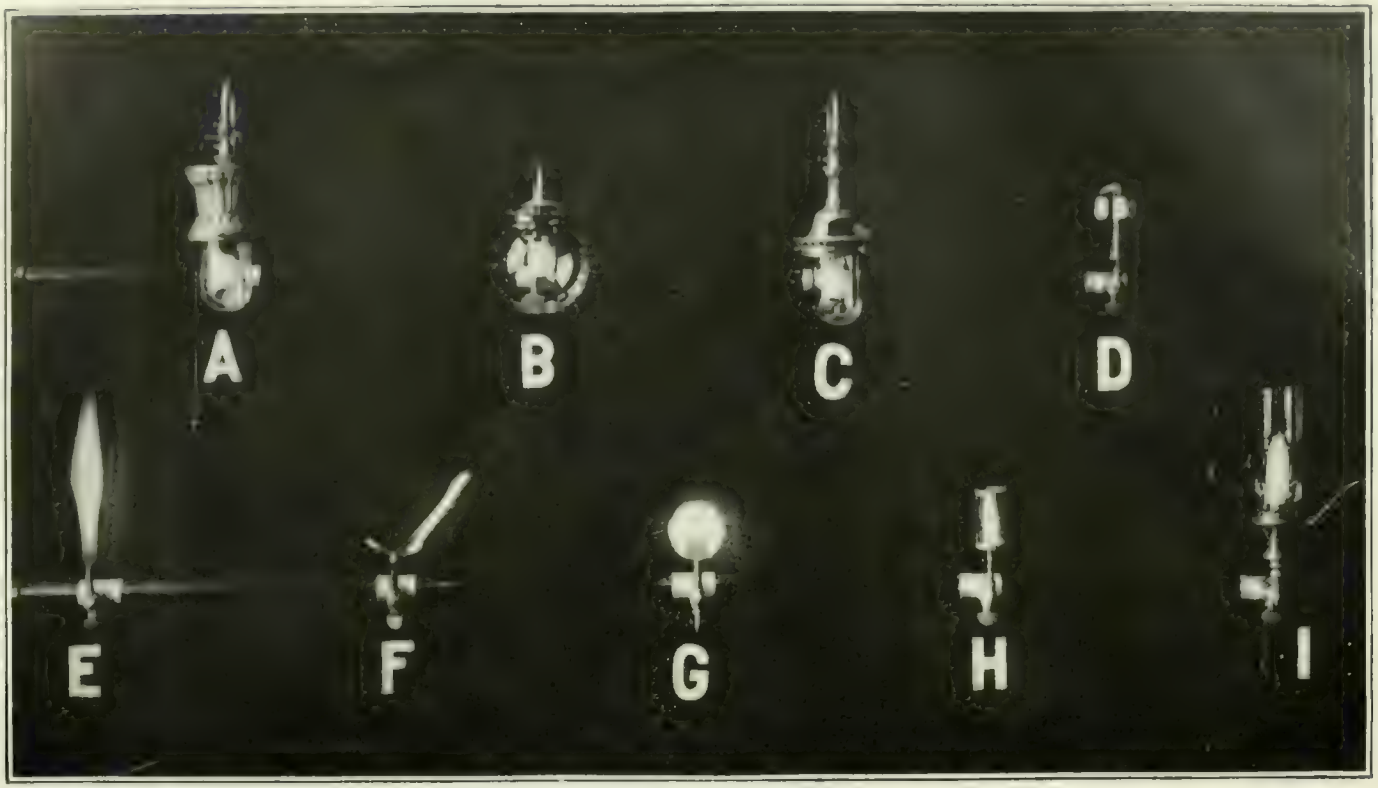


FIG. 1. *Types of small gas-lighting units*

A, high-grade inverted mantle lamp with stack, a recent development; *B*, cheaper type of inverted mantle lamp, has no thermostat and only crude gas regulation; *C*, usual type of high grade inverted mantle lamp with thermostat and good gas regulation; *D*, C. E. Z. mantle lamp, *E*, open-flame burner, with lava tip missing; long, smoky flame is easily blown about by drafts and may easily set fire to surroundings; a burner in this condition is not only dangerous but extremely inefficient; *F*, open flame burner with broken or dirty lava tip causing deflection of flame, likely to set fire to surroundings; the tip is of a type likely to be lost; *G*, well formed open flame; tip is so fastened that it can not be easily broken or lost; *H*, junior upright mantle lamp, with mica chimney; *I*, high-grade upright mantle lamp of usual type with gas-adjustment device

INFLUENCE OF QUALITY OF GAS AND OTHER FACTORS ON THE EFFICIENCY OF GAS-MANTLE LAMPS

By R. S. McBride, W. A. Dunkley E. C. Crittenden, and A. H. Taylor

CONTENTS

	Page
I. Introduction	3
1. Purpose and scope of investigation	3
2. Variables involved	4
3. Conditions of comparison	5
4. Gases used for comparison	7
II. Previous work	8
III. Experimental apparatus and methods	11
1. Lamps and accessories	11
2. Lamp adjustment	11
3. Photometric apparatus	12
4. Accessory apparatus	12
5. Preparation and testing of gas samples	13
6. Recording and computing results	14
7. Difficulties encountered	16
IV. Results of experiments	17
1. Determination of reduction factor	17
2. Pressure of gas	22
3. Gas and air adjustments	24
4. Fitters' adjustment	27
5. Kind of gas	28
6. Fluctuation of gas quality	43
7. Peculiarities of individual units	46
V. General conclusions	47
Appendix.—Data concerning various gases used	48
1. City gas	48
2. Benzolized city gas	49
3. Water gas	49
4. Coal gas	49

I. INTRODUCTION

1. PURPOSE AND SCOPE OF INVESTIGATION

During the past few years there has been a growing tendency throughout the country toward the substitution of heating-value standards of gas supply for the candlepower standards previously in force. In many cases this change in standards has been accompanied by a decrease in the number of heat units per cubic foot

supplied in the gas, and the question has arisen in many cases whether the usefulness of a gas for various purposes is directly proportional to the total heating value of the gas. Claims have been made by some gas engineers that within the limits of heating value of manufactured gas as usually supplied, the gases having the lower heating values can be used with higher efficiency, at least in some appliances, and also with less trouble because of imperfect adjustment of the appliances.

As a part of the general investigation to determine the relative usefulness of gases of different heating values, the Bureau of Standards has experimented with mantle lighting, cooking, water heating, and other appliances, and has made observations of some appliances in actual use on customers' premises. This paper deals with one phase of this question, namely, the usefulness of gases of various qualities when burned under laboratory conditions in certain typical commercial mantle lamps. It also incidentally shows the influence of other factors on the efficiency, candlepower, and satisfaction in use of these lamps. The results of field observations of lamps operating under commercial conditions are given in another paper[1]; and the cooking, water-heating, oven, and other appliance investigations are also to be separately reported.

2. VARIABLES INVOLVED

The efficient operation of a gas-mantle lamp depends upon the proper proportioning of the gas and air entering the lamp in order that the zone of the highest temperature may occur in the comparatively limited space near the mantle. The ordinary low-pressure mantle lamp entrains its own primary air for combustion by the injector action of the entering jet of gas. The amount of air entrained necessarily depends upon the velocity and mass of the issuing gas and these, in turn, are dependent upon the quality of the gas, its pressure, and the size and design of the gas orifice. Mantle lamps are so constructed that the size of the gas or air ports, or both, can be adjusted, and when a lamp has been adjusted to a given set of conditions, any change in these conditions, if not accompanied by a readjustment of the gas and air supply entering the lamp, may have a marked effect upon the operation. A study of the effects of these changes is therefore of first importance; and the control of the variables or the proper allowance for their effects in interpreting the results of any test is

[1] Bureau of Standards Technologic Paper 93, by R. S. McBride and C. E. Reinicker.

essential. A considerable portion of this paper therefore deals with these variables and the effects produced by them.

The efficiency of the use of gas in mantle lighting is largely determined by the following variables, several of which have been investigated more or less completely:

1. Peculiarities of the lamp, as determined by design of the type and by the variations of individual units of a type.
2. Condition of the lamp, especially the freedom from those partial obstructions in gas and air passages which are often very detrimental to efficiency.
3. Quality of the mantle.
4. Character of gas, including composition, density, heating value, etc.
5. Lamp adjustment, both gas rate and air-shutter position.
6. Atmospheric conditions, especially humidity, barometric pressure, and air currents about the lamp.
7. Pressure of gas supply.

It is obvious that some of the above-named variables are under the control of the observer while others can not be readily controlled; but the effects of all have to be considered. The difficulty of control of some of the variables, even under laboratory conditions, and the complexity of their effects in many cases, make the investigation of mantle lamps necessarily less precise than the investigation of types of apparatus in which all the conditions are under control. There is still much to be learned about the conditions affecting lamps, and only after a very extended study and a standardization of lamps and accessories could the operation of lamps under a given set of conditions be precisely predicted and all the present irregularities of operation be explained. While at times these irregularities have been very annoying, it is believed that, in general, their effect has been sufficiently eliminated in this work to make the comparisons reliable within the limits of accuracy attainable.

3. CONDITIONS OF COMPARISON

The efficiency of a lamp—that is, the output of light per unit of heat liberated by the gas in a given time—is different under different conditions. Hence, in comparing the efficiencies of operation of a lamp supplied with different gases the conditions for comparison must be specified. The efficiency of a mantle lamp is usually expressed as the total output of the light, measured in lumens, per unit of heat supplied per hour; that is, lumens per

Btu per hour. This is equivalent to expressing the efficiency as quantity of light produced per heat unit supplied, or lumen-hours per Btu.[2]

All other conditions remaining the same, the gas and air supply to a lamp may be adjusted to give (*a*) maximum candlepower, (*b*) maximum efficiency, or (*c*) some arbitrarily chosen result depending upon local conditions. By efficiency at maximum candlepower is meant the output of light per unit of heat liberated per hour when the lamp is producing the maximum intensity of light possible under the chosen set of operating conditions, only the gas and air being adjusted to produce that result. By efficiency at maximum efficiency is meant the light output per unit of heat liberated per hour when the gas and air supply are so adjusted that the efficiency is the maximum obtainable under the other prescribed conditions. The condition mentioned in (*c*) presupposes that the air and gas adjustment have been prescribed for some special reason, as, for example, in cases where the adjustment to maximum candlepower would tend to carbon the mantles after a time and consequently the air and gas have been changed from such an adjustment to avoid this. In such instances the manner of adjustment is specified so as to make the condition more or less reproducible.

The adjustment to give the maximum candlepower of the lamp is the condition probably most nearly approached in practice, since in making the adjustment an effort is usually made to secure the greatest brilliancy possible. The eye is able to detect fairly closely the point at which maximum brightness is obtained, but since the unaided eye is not sensitive to very small changes in brightness and since the increasing brilliancy of some portions of a gas mantle is often accompanied by relative darkening of other parts, the true maximum can be obtained with certainty only with the aid of a photometer. By varying the gas and air adjustments and at the same time noting the intensity of the light with a photometer, a point can be reached where any change of adjustment in either direction results in a decrease of light intensity. This condition may be sharply defined in some cases, but in others the change of intensity is so gradual that the adjustments may be varied over a considerable range without perceptibly affecting the intensity of the light, though the efficiency may be changing quite rapidly. It should be noted in this connection

[2] A lumen is the unit of light flux, or output, and is of such magnitude than a light of one mean spherical candle has an output of 4π (12.57) lumens.

that the control of the many variables affecting mantle-lamp operation is so difficult and a lamp is so sensitive to external influences, such as drafts, that the establishing of the true maximum to a high degree of certainty is well-nigh impossible.

The point of maximum efficiency of a lamp is accompanied by no distinctive phenomenon apparent to the observer even when aided by the photometer. Therefore, the efficiency at each observation must be determined by computation from measurements, and a sufficient number of observations at different adjustments must be made so that the maximum can be selected by graphical methods. The significance of these changes will be made more evident by the consideration of Fig. 5, which shows for four units the influence of gas adjustment upon consumption, candlepower, and efficiency. (See p. 25.)

Although not commonly realized in practice, the adjustment to give maximum efficiency is of importance in comparison of various gases and various conditions of supply, since clearly it is desirable that a lamp be operated as nearly as possible at such adjustment. Comparison has been made therefore both for adjustment for maximum candlepower and for maximum efficiency. The relative efficiencies with the different conditions of supply and operation were thus determined by comparing lumens per Btu per hour for the several combinations contrasted.

By fitters' adjustment is meant any arbitrary method of adjustment which is prescribed to accomplish some special result, such as the avoidance of carboning mantles or the attainment of good operation under predicted service conditions which may be somewhat different from the conditions existing at the time of adjustment. For example, a lamp may be adjusted during the daytime for the pressure or other conditions which are expected to obtain at night. This will be further discussed in a later portion of this paper.

4. GASES USED FOR COMPARISON

Since the chief object of the work presented in this paper was to study the operation of lamps with various gases, one of the first problems was to supply the various gases in adequate amounts for testing. It was decided to confine the study to gases typical of those supplied in practice to various communities; and since the Bureau had received special inquiry concerning the relative usefulness of carbureted water gases of about 665 Btu and of 565

Btu, and of coal gas of 565 Btu, these were selected as the typical gases to be studied. For the sake of comparison and further study of certain variables, some specially enriched gases of higher heating value were also used in a few cases. Since typical commercial gases vary from time to time within certain limits, both as to composition and heating value, it was not considered necessary to maintain exactly the same quality of gases throughout the various series of tests; nor was it practicable to do so. An effort was made, however, to prevent as far as possible variations between sets of observations which were intended to be comparable.

II. PREVIOUS WORK

A number of investigators have studied the relation between the heating value of the gas burned and the mantle-lighting efficiency obtained. The problem has been attacked from different standpoints and widely varying conclusions have been drawn. These differences are usually explainable when the details of the investigations are known. In some cases it appears that variables other than the heating value of the gas have exerted such large influences that the effects of the variable in question have been obscured. In other cases the results obtained, while applicable to the conditions of the tests, would not be generally applicable to commercially existing conditions. The following is a summary of the more important of these investigations which have come to our attention:

In 1900 H. Bunte [3] reported to the International Gas Congress at Paris that from his studies he had found that gases of widely different heating values gave practically the same amount of light per cubic foot when used in mantle burners. However, no adjustment was made for gas and air and the burner was designed for gas of comparatively low heating value.

In 1902 A. H. White, H. Russell, and A. F. Traver [4] reported that other conditions being the same, the amount of light given per cubic foot by a gas when used in an incandescent gas burner (adjusted to give maximum candlepower at 2 inches gas pressure in each case) was proportional to the net heating value and increased at the rate of one candle for each increase of 4 calories (15.87 Btu) in the heating value per cubic foot. In their experiments they

[3] Bunte, Mayer, and Teichel, J. fur Gasbeleuchtung, 51, p. 265; 1908.
[4] Am. Gas Light Jour., 76, p. 413; 1902.

used a "C" upright Welsbach burner and a Welsbach mantle. They stated that the point of maximum efficiency coincided with the point of maximum candlepower in all cases. The gases tested by them ranged in quality from 246 to 1050 Btu per cubic foot and included carbon monoxide, hydrogen, blue water gas, carbureted water gas, coal gas, natural gas, Pintsch gas, and various mixtures of gases. They found that whereas the natural gas and Pintsch gas, especially the former, varied quite widely from the general rule, those gases representing the usual qualities supplied by gas companies followed the rule very closely.

In 1903 St. Claire-Deville [5] of the Paris gas works reported that his work showed that the lighting effect produced by gas used in incandescent mantle burners was, apart from differences in mantles, proportional to the net heating value of the gas, providing the gas was burned in each case with the amount of air suited to it. He found it necessary, however, to supply air under pressure for the perfect combustion of some of the very rich gases.

In 1908 Max Mayer and H. Schmidt [6] discussed the work of St. Claire-Deville and presented data to show that while Deville's conclusions established a useful rule for judging the value of a commercial illuminating gas for mantle lighting, they did not apply in all cases to the behavior of special mixtures of illuminating gas with hydrogen or with carbon monoxide, even though burned with the same rate of heat liberation, equal flame volume, equal flame temperature, and the same form of flames, on account of the degree of dissociation of carbon dioxide when gas mixtures producing large proportions of carbon dioxide in their combustion products are burned.

In 1909 Arthur Forshaw [7] reported some work done by him at the University of Leeds on the comparative efficiencies of hydrogen and carbon monoxide when burned in mantle burners. He found that burned in an ordinary atmospheric burner, carbon monoxide gave a much higher "duty" than hydrogen and concluded that the illuminating efficiency of a mantle burner is not necessarily proportional to the heating value of the gas.

Max Mayer [8] reported in 1910 some investigations in which he took samples of the gas and air mixtures from the mixing tube of an upright mantle burner and studied the gas-air ratio in each case in connection with the luminous efficiency of the burner.

[5] Jour. Gas Lighting (London), **83**, p. 499; 1903.
[6] Jour. für Gasbeleuchtung, **51**, p. 1137; 1908.
[7] Jour. Gas Lighting (London), **106**, p. 865, 1909.
[8] Jour. für Gasbeleuchtung, **53**, p. 933, 1910.

He concluded that the composition of the gas and its velocity of efflux play almost a greater part than does the quantity of air drawn in, and that as the heating value falls and the proportion of heavy hydrocarbons decreases the efficiency increases in mantle lamps which entrain their own air at low pressures. He also concludes that the higher the percentage of hydrogen becomes the higher is the efficiency. Finally he concludes that while St. Claire-Deville's conclusions are within a few per cent applicable to the usual commercial illuminating gases, they would not be applicable to gases of higher or lower heating values in ordinary low-pressure burners. He criticised Forshaw's conclusions upon the ground that owing to the greatly differing properties of hydrogen and carbon monoxide they could not be fairly compared with the same burner.

In 1915 R. F. Pierce[9] reported some observations of the behavior of mantle lamps with various water gases. The tests were performed with an upright lamp and "were planned with a view to demonstrating that calorific value, specific gravity, and gas candlepower do not definitely specify a gas for commercial purposes," but that the composition of the gas and the flame temperature which is will produce are the determining factors. Pierce presented tables and curves to substantiate his position and from these concluded that his a priori statement was proved. He drew the far-reaching conclusion that neither gas candlepower, specific gravity, or calorific value, are of the slightest value in predetermining the performance of a lamp or in fixing standards of service for gas lighting. However, one of us[10] reviewed Pierce's work and showed that he was not justified in drawing the conclusions which he did from his own data. It was demonstrated that if Pierce's results were properly grouped—for example, for each 20 Btu interval of heating value of the gas—these same data would prove for gases within the commercial ranges of heating value now existing, just the opposite of Pierce's conclusions. In other words, they show that for commercial ranges of heating value the usefulness of the gas for mantle lighting is directly proportional to the gross heating value of the gas.

---

[9] Am. Gas Light Jour., 103, p. 1, 1915.

[10] R. S. McBride, Am. Gas Light Jour., 103, p. 353, 1915.

III. EXPERIMENTAL APPARATUS AND METHODS

1. LAMPS AND ACCESSORIES

The lamps used in the work discussed in this paper comprise the following higher-priced units of Welsbach make: No. 6 Reflex, No. 71 Upright, Junior, and C. E-Z; tests were also made with the Welsbach No. 7 inverted lamp and some lamps of the upright and junior types purchased in 5-and-10-cent stores. Some of the lamps tested are shown in Fig. 1. All the lamps were practically new and were not changed in any way, except that one Junior lamp was provided with an accurately centered needle valve, so as to make the distribution of the gas mixture within the mantle more symmetrical.

In making photometric tests with the various lamps the only forms of glassware used were the plain cylinders for the Reflex and upright lamps. The regular mica chimney was used with the Junior, and the C. E-Z lamp was used without any glassware whatever. When candlepower was to be taken in one direction only, the lamps were set up before a black background so as to avoid errors from reflected light. The lamps were also shielded from drafts of air, but not sufficiently to interfere with proper ventilation. It was found necessary in most cases to keep all windows closed, as it was found that all the lamps were very sensitive to air currents even though shielded fairly well. In all tests of upright and inverted lamps regular mantles as supplied on Government contracts were employed; for Junior and C. E-Z lamps the mantles furnished by the makers of the lamps were used.

2. LAMP ADJUSTMENT

After setting up the lamp to be used it was lighted and allowed to burn, under ordinary conditions, for at least one-half hour before any observations were made. This was important since it was found that a lamp, even with a mantle which had been used several hours, did not reach its maximum candlepower at any adjustment until several minutes after lighting. If the mantle was a new one, a longer period was always allowed.

When the conditions of gas supply for the test had been established, the adjustment of the lamp was made by one observer while another read the photometer and kept the conditions external to the lamp constant as far as possible. In adjusting a lamp

the gas screw or other regulating device was manipulated until the desired condition was reached as judged by the photometer or by the gas rate if the latter was the determining factor in the adjustment. The air shutter was then manipulated until its position to give maximum intensity for this gas rate was reached. In general, the air shutter was wide open; and in the later sections of this paper when no mention is made of this detail the shutter may be understood to have been so placed.

3. PHOTOMETRIC APPARATUS

Several photometers with their accessories were used in this work. In all cases the comparison was made between the gas lamp and an electric standard of known value; the difference in color in all cases was compensated for by a color cell or glass placed in the path of the light from the electric lamp to the photometer. In some of the runs the lamp was placed in an 88-inch integrating sphere and the mean spherical candlepower measured directly. In most cases, however, the intensity of the light in only one direction was determined; and the mean spherical candlepower when needed was computed by applying suitable reduction factors. The candlepower of upright-mantle lamps was observed in the horizontal direction and of inverted and C. E-Z units at an angle of 20° below horizontal. Unless otherwise qualified the term "candlepower" as used in this paper refers to the intensity in these directions.

The other photometers used in such comparisons included a standard model Sharp-Millar portable photometer and a Schmidt and Haensch (Bechstein) universal photometer. Each of the photometers was equipped with suitable rheostats and volt or millivolt meters to keep the intensity of the comparison lamp constant. The comparison lamps were calibrated at suitable intervals against standard lamps.

4. ACCESSORY APPARATUS

The gas rate was measured in each case by a calibrated laboratory wet test meter timed by a stop watch, correction being made to standard conditions of temperature and pressure.

Gas pressure was measured by a water manometer and the pressure during a given run was kept constant either by a wet pressure regulator or by a dry photometer governor.

A mercury barometer, Assman psychrometer, and U. G. I. dew-point apparatus were used to determine the atmospheric pressure, atmospheric humidity, and the oil and water dew points of the gas, respectively.

5. PREPARATION AND TESTING OF GAS SAMPLES

The city gas available for use in testing at this Bureau was a mixed water and coal gas of about 650 Btu per cubic foot. In order to make gases of other qualities, resembling typical water and coal gas, the city gas was used as a basis and varying proportions of the constituents were added to obtain the desired composition. For the manufacture of a gas resembling straight water gas it was necessary to add carbon monoxide and hydrogen in proper amounts to the city gas; while in producing a gas resembling coal gas it was necessary to add methane, hydrogen, and carbon dioxide to the city gas. The carbon monoxide was obtained by the decomposition of sodium formate with sulphuric acid, the hydrogen was produced electrolytically, and the carbon dioxide was commercial material available in cylinders under pressure. The methane was obtained in the form of natural gas from a gas field in western Pennsylvania, very rich in methane; it was shipped to us in cylinders at about 275 pounds' pressure.

It was not found practical to bring the synthetic mixture to exactly the same composition in each case; but having found the average composition of the city supply from a number of analyses, it was possible to approximate very closely the desired composition and also to obtain the Btu desired. The usual procedure was to introduce the estimated amount of each special gas into a gas holder of 30 cu. ft. capacity and then to fill the tank to the limit of its capacity with the city gas.

After making up the sample it was allowed to stand several hours to become thoroughly mixed, or in cases where convenient the gas was forced back and forth several times from one tank to another to hasten the mixing of the sample. The heating value and in some cases the open-flame candlepower and specific gravity were then determined and a sample of the gas was taken for analysis.

While the composition of the various gases varied from day to day, the data of Table 1 are representative analyses, being averages of many of each type of gas. (See Appendix.)

TABLE 1.—Typical Gas Analyses

	City gas (mixed)	Water gas	Coal gas	Enriched [illegible]
CO_2	4.1	4.4	5.1	3.5
O	.6	1.0	1.3	.3
Illuminants	10.1	6.9	3.9	13.4
CO	19.7	26.2	7.0	19.1
CH_4	23.9	18.9	30.0	25.3
H	35.9	38.5	47.1	34.7
N	5.7	4.1	5.6	3.6
Btu average	650	550	570	740
Candlepower	18	7	Below 7	25
Sp. grav	.61	.60	.....	.64

The heating value of each sample of gas used was carefully determined; but since it was found that the city gas gave a fairly uniform analysis over considerable periods, analyses of that gas were made less frequently than of the synthetic water and coal gases, practically all of which were analyzed. As may be noted from the analyses, the city gas is about 80 to 85 per cent water gas, the remainder being coal gas. The city gas showed variations in heating value through the period of the tests from 618 to 686 Btu, the water gas from 487 to 590, the coal gas from 540 to 592, and the benzol-enriched city gas from 697 to 781 Btu. However, the heating value of the water gas used in tests for studying the direct comparisons of operation of city and water gas in lamps was always about 560 to 565 Btu, while the average values of the other gases were about as given in the table above.

6. RECORDING AND COMPUTING RESULTS

For convenience of presentation in this paper the work done has been divided into series. A series included all the experiments made to show the effects of a certain variable condition upon the operation of the various lamps. The work of a series extended in some cases over a period of several days and was divided into runs. A run was usually the work of one day or in some cases only a few hours. During a single run a single condition was varied and all other operating conditions were kept constant, or if this was impossible any changes were observed and recorded so that their effect could be corrected. A run included several sets of observations, all of which were comparable with each other. In some cases two or more runs were made the same day under conditions which made the runs comparable. A single set of observations

included the data obtained for each single value of a variable, all other conditions remaining constant. Table 2 shows a typical data sheet for a single run.

TABLE 2.—Typical Data Sheet

Series No., 1; Run No., 1; Sheet No., 1; Date, July 19, 1916; Lamp, No. 6 Reflex; Air shutter, open; Fixture cock, open wide; Gas No., 34; Btu, 650; Photometer factor, glass in, 23.13, out 5.0; Meter temp., 70; Bar. (corr.), 29.57; pressure at meter, 5 in.; Factor, 0.970; Humidity: Dry bulb, ——; Wet bulb, ——; L. per cubic meter, ——.

Set No.	Gas pressure, inches	Adjustment pressure, inches	Gas rate			Photometer readings						Candlepower	Candles per cubic foot
			Time, seconds per revolution	Cubic feet per hour (uncorrected)	Cubic feet per hour (corrected)	1	2	3	4	5	Average		
[a]1	1	3	146	2.5	2.4	3.19	3.10	3.10	3.26	3.19	3.17	15.8	6.6
2	1½	3	112	3.2	3.1	2.03	2.02	2.02	2.09	2.08	2.05	47.4	15.3
3	2	3	96	3.7	3.6	2.70	2.74	2.70	2.70	2.71	2.71	62.7	17.2
4	2½	3	87	4.1	4.0	2.90	2.93	2.96	2.95	2.90	2.93	67.8	16.9
5	3	3	80	4.5	4.4	3.10	3.12	3.13	3.13	3.19	3.13	72.4	16.6
6	3½	3	74	4.9	4.7	3.42	3.40	3.40	3.41	3.42	3.41	78.9	16.7
7	4	3	70	5.1	5.0	3.60	3.61	3.67	3.72	3.68	3.66	84.7	17.0
8	4½	3	66	5.4	5.3	3.40	3.40	3.41	3.39	3.40	3.40	78.6	14.9
9	5	3	60	6.0	5.8	3.42	3.40	3.35	3.39	3.27	3.37	78.0	13.4

[a] Glass out; all others, glass in.

In computing the results, the gas rate per hour for each set was computed from the time elapsed during a meter revolution, the volume being corrected for temperature and barometric pressure in the usual manner. The average candlepower for each set was usually obtained by averaging all the photometric observations for each set and multiplying this average by a factor which converted the photometer reading to spherical candles. This factor took into account the constant of the photometer and the reduction factor for converting the candlepower in the direction observed to mean spherical candles. In most cases where the same gas was used throughout a run, the efficiency was expressed simply in candles per cubic foot per hour; the candlepower observed in a given direction was used, since the reduction factor to mean spherical candlepower had been found to be practically constant with different adjustments. In some cases the efficiency was further expressed in lumens per Btu per hour, determined by multiplying the mean spherical candlepower by 12.57 (4π) to get lumens, and then dividing by the Btu per cubic foot of gas times the rate of gas consumption per hour.

7. DIFFICULTIES ENCOUNTERED

The difficulties encountered in this work were chiefly caused by external influences or by peculiarities of the individual lamps tested. Some of these difficulties are here mentioned to aid others in similar work.

As has been mentioned before, it was found that the lamps were very sensitive to drafts. While this would not necessarily be of consequence in commercial operation, it was very annoying where the effects of small variations of operating conditions were to be determined. It was found necessary to work in a room free from drafts and as an added precaution to shield the lamps by screens.

Another factor which had to be considered was the lag in the candlepower change caused by changing conditions. Not only was it necessary to delay making observations after first lighting a lamp, but it was also found that changing the pressure or gas adjustment was followed by a gradual adjustment of the lamp to the new conditions and it was necessary to wait until the operation of the lamp became steady.

Some of the most annoying difficulties were caused by imperfections of lamp construction. For example, it was found that with some of the lamps tested the stream of gas-air mixture entering the lamp was not symmetrical with respect to the mantle, and changed shape as the gas adjustment was changed. Changes in the gas adjustment resulted in such cases in an erratic variation, not only in the relative brightness of different sides of the mantle, but also in the total light output. This peculiarity was noticeable in the upright and Junior types of lamps, especially the latter. While this irregularity might have little importance in the commercial use of the lamp, it was troublesome in this work and was only remedied by substituting in the lamp a new adjustment screw accurately fitted and symmetrical.

Another condition encountered which was not serious but which delayed the work on certain occasions was the difficulty of predicting the increase in rate of gas which would result from a change in the gas adjustment. It was found that the effect of the gas-adjusting screw or other device varied greatly with different positions of the adjustment. A small fraction of a turn in some parts of the range would cause a much greater change in the operation than one or two complete turns in other parts.

IV. RESULTS OF EXPERIMENTS

In this work, as in all gas photometry, a large number of series of tests were needed in order to establish the various conclusions; but in this paper it is desired to set forth only the conclusions and a few data typical of the numerous series carried out to establish the magnitude and direction of influence of the various factors. It should be borne in mind in considering these data that the absolute values given should not be taken as of particular significance, since in many cases they depend upon the atmospheric conditions or the methods of testing arbitrarily chosen to bring out certain particular information; and furthermore, these values would not always be reproducible with all lamps of a type even if the working conditions were duplicated. However, these differences have, in general, been wholly eliminated from influence upon the qualitative conclusions by careful control within the period of test, of the factors that might affect the particular comparison desired; and it is believed that the results given show the influence of the variables in a way typical of all lamps of the types tested.

1. DETERMINATION OF REDUCTION FACTOR

In order to facilitate the testing of mantle lamps it was necessary to make a large number of the photometric measurements with apparatus by which the intensity of the light in one direction only could be measured. In order that these measurements in a single direction might show directly the variation in the total light output from the lamp, a direction was chosen in which the intensity bore a constant relation to the mean spherical intensity under changing conditions.

A distribution photometer was used to determine the value of this reduction factor; that is, the factor by which the candlepower in one direction should be multiplied in order to determine the mean spherical candlepower. These experiments confirmed the previous conclusions of others; the results are presented here largely because of the possible interest in some of the data for the inverted and C. E-Z units.

During the measurements the lamp was so screened that only the light given off in a certain direction was reflected to the photometer by two plane mirrors. The position of these mirrors in a vertical plane relative to the lamp could be adjusted, and by

successive observations a series of values was obtained representing the intensity at different angles from a point directly below the lamp to a point directly above it. By plotting the candlepower values from these observations the distribution curves of the lamp under each operating condition were obtained.

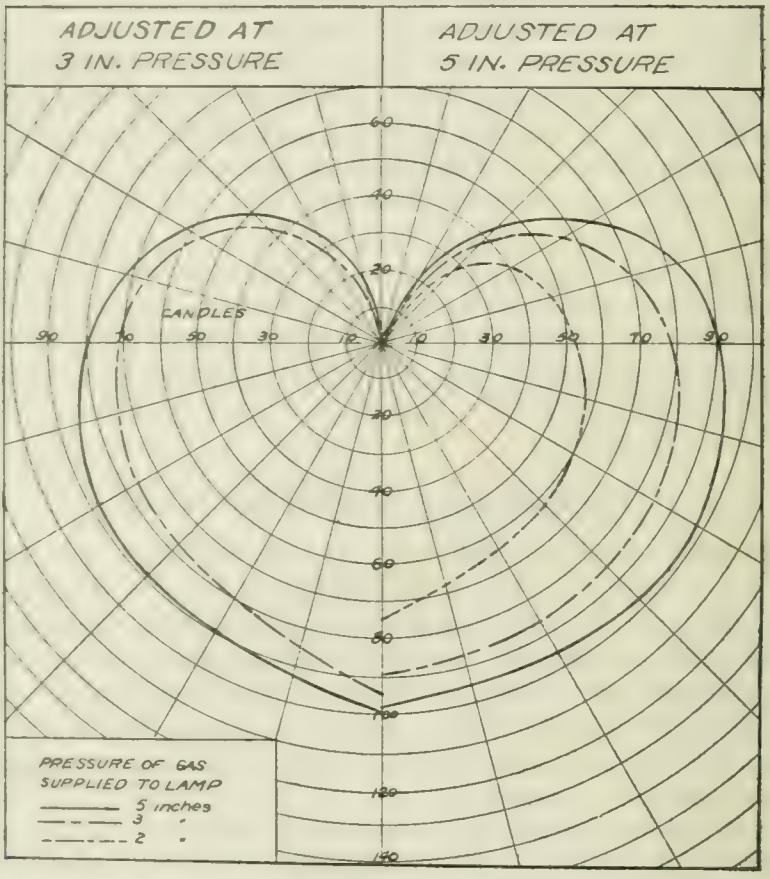


FIG. 2.—*Distribution of No. 6 Reflex lamp in a vertical plane adjusted to maximum candlepower at 3 and 5 inches gas pressure and operated at the pressures indicated*

The curves of Figs. 2 and 3 show the distribution of light determined by the distribution photometer for the No. 6 Reflex and the C. E-Z lamps. The curves on the right half of Fig. 2 show the distribution of light when the lamp viewed in a direction 20° below the horizontal was adjusted to give maximum candlepower at 5 inches pressure with mixed gas of 636 Btu, when operated at the various pressures indicated. Similarly the

curves at the left of the vertical axis give the distribution when the lamp was adjusted at 3 inches pressure.

The curves of Fig. 3 are for the C. E-Z lamp adjusted at 5 inches pressure for maximum candlepower at 20° below horizontal with mixed gas of 634 Btu per cubic foot, when operated

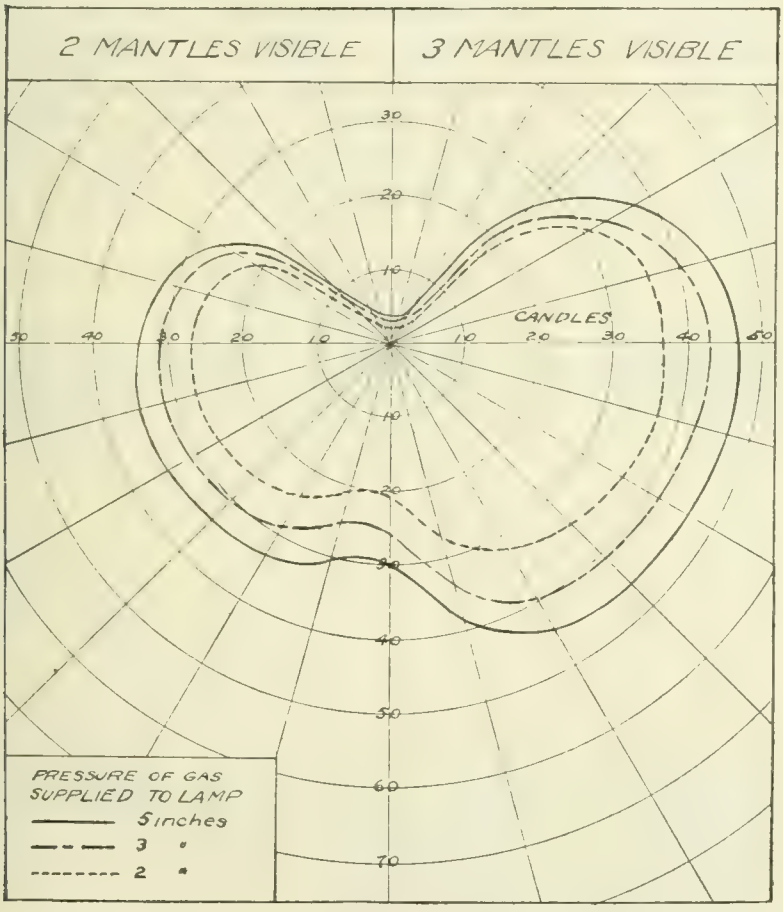


FIG. 3.—*Light distribution of C. E-Z lamp in a vertical plane adjusted to maximum candlepower at 5 inches gas pressure and operated at the pressures indicated*

at the pressures shown. The curves show the difference in candlepower when two or three mantles are visible and make clear the lack of uniformity in the light distribution. To determine these values for the C. E-Z unit the right and left mirrors of the photometer were used alternately, the one not in use being covered with a black cloth.

To determine the reduction factor from these data the mean spherical candlepower of the lamp under the given conditions was computed by multiplying the intensity at each 10° interval by the factor representing the ratio of the area of a zone measured 5° each side of that angle to the total area of a sphere. The sum of all the products thus obtained gave the mean spherical candlepower of the lamp. It is obvious that zones of surface which are bounded by small circles—that is, those near the vertical axis—have the smallest area and consequently are of least importance in the calculation. The most important directions, in which the intensities should be carefully established, are those near the horizontal. The following tabulation (Table 3) illustrates the use of such data for the Reflex lamp.

TABLE 3. Computation of Mean Spherical Candlepower of Reflex Lamp

Zone measured angle from point directly beneath lamp)	Spherical-candlepower constant (relative area of zone) A	Candlepower B	Weighted candlepower (A times B)	Zone measured (angle from point directly beneath lamp)	Spherical-candlepower constant (relative area of zone) A	Candlepower B	Weighted candlepower (A times B)
[illegible]	.002	98	0.2	100°	0.086	86	7.4
[illegible]	.015	96	1.5	110°	.082	78	6.4
[illegible]	.030	96	2.9	120°	.075	64	4.9
[illegible]	.044	96	4.2	130°	.067	50	3.4
[illegible]	.056	98	5.5	140°	.056	36	2.0
[illegible]	.067	98	6.6	150°	.044	22	1.0
[illegible]	.076	98	7.4	160°	.030	12	.4
[illegible]	.082	97	8.0	170°	.015	[illegible]	.1
[illegible]	.086	94	8.1	180°	.002	0	.0
[illegible]	.087	91	7.9	Total, mean spherical candlepower=			77.9

This method of computation of mean spherical candlepower was applied to the data for the C. E-Z and the Reflex lamps and then the ratio of the mean spherical candlepower and the candlepower in various directions was computed. The results of two such series of computations are shown in Table 4.

These data show that for the Reflex lamp there is little choice between measurements at any angle from the horizontal to 50° below horizontal, since the reduction factor under different conditions of operation appears to be approximately constant at any of these angles. With the C. E-Z lamp the reduction factor appears to be nearly constant for angles about 20° to 30° below horizontal. These conclusions confirm the work done by others which indicated that for observations at about 20° below horizontal the reduction factor was substantially constant. This angle was

also conveniently obtained with the apparatus which was at hand, and therefore throughout later work observations of the Reflex and the C. E-Z lamps were made in this direction.

Similar work was done on the upright mantle and Junior lamps. This showed a constant value for the reduction factor from horizontal candlepower to mean spherical candlepower. This is to be expected on account of the shape of the mantles.

TABLE 4.—Reduction Factor

REFLEX NO. 6 LAMP

Angle below horizontal	Lamp adjusted at 5 inches pressure and operated at—			Lamp adjusted at 3 inches pressure and operated at—		Average value	Maximum deviation from average
	5 inches	3 inches	2 inches	5 inches	3 inches		
Horizontal	0.854	0.885	0.914	0.912	0.897	0.892	0.038
20° below horizontal	.801	.791	.812	.852	.820	.815	.037
30° below horizontal	.792	.772	.785	.835	.811	.799	.036
40° below horizontal	.792	.767	.765	.819	.801	.789	.030
50° below horizontal	.797	.770	.747	.803	.797	.783	−.036
60° below horizontal	.809	.771	.724	.789	.797	.778	−.044
C. E-Z LAMP							
Horizontal	0.827	0.794	0.802	0.794	0.774	0.798	0.029
20° below horizontal	.804	.788	.798	.772	.789	.790	−.018
30° below horizontal	.815	.808	.809	.790	.830	.810	−.020
60° below horizontal	.844	.851	.905	.816	.926	.868	−.058

The absolute value of the reduction factor is of no great importance in any of the work of the following sections since in practically all cases only the relative values on each lamp were of consequence. Nevertheless, in several instances it was found convenient to convert the observations to mean spherical candlepower. The results given above for the Reflex and C. E-Z lamps indicate that the average for the former is slightly above 0.80 and for the latter slightly below this figure. The work of others indicated that 0.80 was the correct factor, and therefore we have employed this for both the Reflex and the C. E-Z lamps. For upright-mantle lamps we have used the factor 0.75, basing our choice not only upon our own work but also upon results of several previous workers. It is desired to emphasize again that the absolute value of these factors is of no consequence since in most of our work the conclusions might about as well have been reached by use of the candlepower in the single direction for which we had shown the factor to be practically constant.

2. PRESSURE OF GAS

The effect of the variation of pressure on the operation of mantle lamps was studied by keeping all other variable conditions as constant as possible and making a series of photometric observations with the supply to the lamp at various pressures. In the tests of a particular lamp the quality of gas was kept uniform by supplying the lamp from a small holder containing about 30 cubic feet of gas, the quality of which had been determined. Each lamp was tested at three adjustments, namely, the positions of the gas adjustment screw (air shutter open) giving maximum candlepower as determined by a photometer, at 2 inches, 3 inches, and 5 inches pressure, respectively. After adjusting the lamp to such maximum candlepower at one of these pressures, the pressure of the supply was varied by steps of one-half or 1 inch from the lowest to the highest pressure under which the lamp would probably be operated in general service. The extreme range of pressure was from one-half to 8 inches for each gas pressure, the candlepower of the lamp and the rate of gas consumption, as well as any peculiarities or irregularities of operation, were observed.

The results of one such series of operations (designated as series 1) are shown graphically in the typical curves of Fig. 4. Since the tests of the various lamps were made on different days, the data obtained from one lamp are not to be compared with the data from another lamp, so far as absolute values are concerned. The various curves for each lamp are, however, comparable and show the shapes of curves typical of each lamp.

The more important conclusions regarding the effects of pressure changes upon the operation of the various lamps tested are as follows:

(*a*) The lower the pressure at which a lamp is adjusted to maximum candlepower, the wider is the opening of the gas adjusting valve, and consequently the greater is the gas consumption at any given pressure.

(*b*) For any adjustment of a lamp, the following results are obtained with changes of the pressure of the gas supplied (from the lowest pressure at which the lamp will remain lighted):

1. The gas consumption is roughly proportional to the square root of the pressure.

2. The efficiency increases up to a maximum with increasing pressure, after which it decreases with further increase of pressure.

3. The candlepower increases with increasing pressure up to and beyond the pressure for the maximum efficiency until a maximum candlepower is obtained, after which the candlepower decreases with a further increase of pressure. However, with

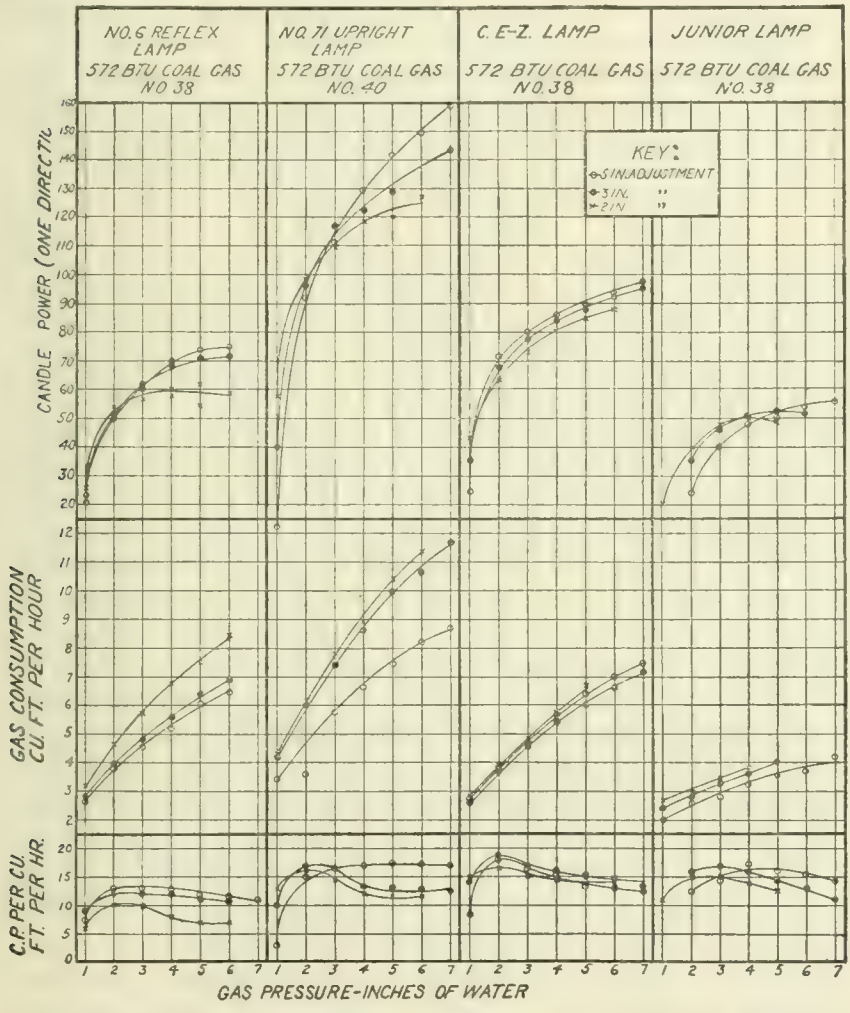


FIG. 4.—*Effect of gas pressure variation upon the efficiency, gas consumption, and candlepower of various lamps*

some of the lamps tested it was not practicable to increase the pressure sufficiently to reach a true maximum of candlepower; this was especially notable with the upright-mantle lamp and to a lesser degree with the C. E-Z lamp.

(*c*) The lower the pressure at which a lamp is adjusted to give maximum candlepower, the greater is the opening of the gas-

adjusting valve, the larger is the gas consumption at a given pressure, and the nearer to each other are the pressures for production of maximum efficiency and maximum candlepower.

(b) When clean and with the air shutters wide open, none of the lamps show any tendency to carbon the mantles under any of the pressure conditions tried.

(c) Regardless of the pressure at which the lamp has been adjusted to give maximum candlepower, the maximum efficiency is almost invariably found at pressures between 2 and 4 inches.

3. GAS AND AIR ADJUSTMENTS

As has already been explained, in comparing results obtained with different gases at a given pressure two definite conditions were used, one being the condition which gave the highest candlepower attainable with the particular lamp and the other that which gave the highest efficiency. In order to obtain these conditions the flow of gas was varied step by step by means of the adjusting device provided in all the lamps, and then (in those lamps which had variable air ports) at each setting of the gas orifice the air ports were set by trial at the position which gave the best performance. The typical curves shown in Fig. 7 were obtained in this way, and similar curves were made for all other comparisons of different gases.

This procedure amounted to a study of the effects of gas and air adjustment under all the variations of other conditions used, but to discuss these effects in detail would require more space than it is desirable to give, and consequently only the results obtained at the maxima of candlepower and of efficiency have been considered.

In plotting these curves the rate of consumption of gas was taken as the basic variable, and as this was regulated by the position of a screw of some kind in each lamp, it is of some interest to show how the consumption varied with position of the screw. The results of a series of observations made for this purpose are shown in the curves of Fig. 5. In making these observations the gas pressure, gas quality, and air-shutter position were kept constant, while the position of the gas screw was varied. At each screw position the candlepower and rate of gas consumption were determined.

It will be noted that in only one case was there anything approaching proportionality of apparent opening to the real opening as shown by the rate of flow of gas. In two of the lamps the orifice was practically wide open when the screw had been moved

over about half of its range, and consequently turning it farther had no effect. In both of these lamps (the Reflex and the Junior) this wide-open position gave the best performance, but this would not be true for richer gases.

The sharply defined maximum in the efficiency curve for the C. E-Z lamp is corroborated by other measurements on this type of lamp.

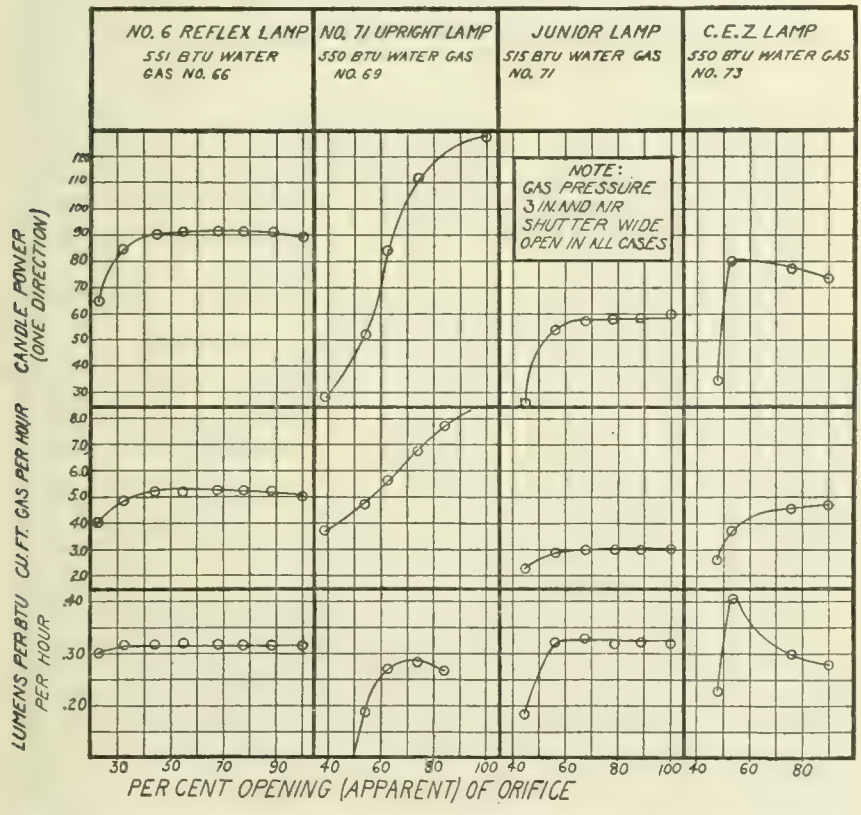

NO. 6 REFLEX LAMP
551 BTU WATER GAS NO. 66
NO. 71 UPRIGHT LAMP
550 BTU WATER GAS NO. 69
JUNIOR LAMP
515 BTU WATER GAS NO. 71
C.E.Z LAMP
550 BTU WATER GAS NO. 73
NOTE:
GAS PRESSURE 3 IN. AND AIR SHUTTER WIDE OPEN IN ALL CASES
CANDLE POWER (ONE DIRECTION)
CU. FT. GAS PER HOUR
LUMENS PER BTU PER HOUR
PER CENT OPENING (APPARENT) OF ORIFICE

FIG. 5.—*Effects of gas adjustment upon candlepower, gas consumption, and efficiency of various lamps*

In this, as in the preceding series, the absolute values obtained with the different lamps are not comparable, because the lamps were tested on different days and with different gases.

The effects of changing the air adjustment depend, of course, on the other conditions of operation. It was usually found that with small gas consumption, and particularly with the leaner gases, the best performance was obtained with the air ports closed as far as was possible. In some such cases it was necessary to

close the ports to prevent the flame from flashing back into the mixing tube. With higher consumptions and richer gases the best performance was obtained with air ports wide open; in fact, this was true over the greater part of the range of desirable operating conditions. At fairly high consumptions, even of lean gases,

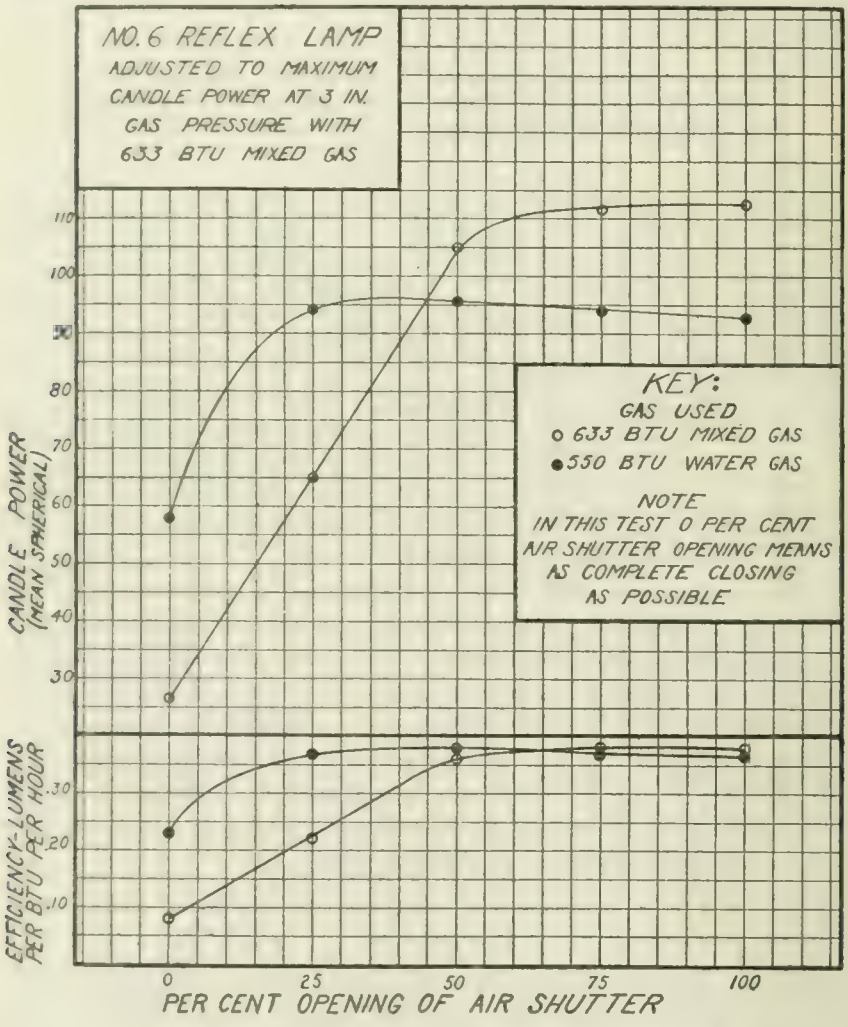


FIG. 6.—*Effect of air shutter position*

there is very little to be gained by closing the air ports, while under some conditions closing them may greatly decrease the efficiency. The air ports should nearly always be wide open.

To illustrate the effects of air adjustment there are plotted in Fig. 6 two series of measurements on a Reflex lamp in which

the gas pressure and gas orifice were kept constant, while the air shutter was set at different positions. The lamp was initially adjusted to give maximum candlepower with mixed gas of 633 Btu per cubic foot with the air shutter wide open, and no change was made except in the position of the air shutter. The consumption was therefore constant with each gas, being 4.7 cubic feet per hour of the 633 Btu mixed gas and 4.6 of the 550 Btu water gas. From relations between consumption, maximum candlepower, and maximum efficiency which will be discussed later, it may be inferred that the initial adjustment gave very close to maximum efficiency for the 550 Btu water gas.

The lamp having been adjusted for a rather high consumption, the candlepower and efficiency fell off rapidly as the air shutter was closed when burning the richer gas, but with the leaner gas there was a slight improvement obtained by partly closing off the air.

In general, the greatest excess of air that can be obtained with any of these lamps has an effect much less serious than the effect of even a slight deficiency of air. Consequently with the leaner gases, which require less air, the lamps are less sensitive to air-shutter adjustments.

Any maladjustment of air shutter with rich gas almost invariably gives too little primary air and hence produces a loss in efficiency and a likelihood of carboned mantles. With lean gases the air shutter can often be nearly closed without great loss of efficiency and there seems to be less tendency to carbon the mantle. The difficulty with the leaner gas is that there is greater tendency of the lamps to back fire and burn in the mixing tube because the shutter is too wide open. However, this has been regarded as a less serious difficulty than that of carboning the mantles because it is a condition that is at once noted by the user and is therefore likely to be corrected promptly by partly closing the air shutter.

4. FITTERS' ADJUSTMENT

In adjusting a lamp for service it is usual to regulate the gas supply so that the maximum candlepower, as judged by the eye, is obtained. However, one very large gas company in the United States has adopted a slightly different adjustment for the lamps which it maintains. The fitters employed by this company are instructed to adjust the gas supply until the candlepower is apparently at a maximum and then to decrease the gas supply

until a slight but distinct diminution in the candlepower is effected. The object in so adjusting is to diminish the probability of carboning the mantles. As will be noted from Fig. 4, the point of maximum efficiency for a given lamp occurs at a lower gas consumption than the point of maximum candlepower, therefore in reducing the gas consumption slightly below the maximum-candlepower point the lamp is actually brought nearer to its point of maximum efficiency. In the course of our work a number of fitters' adjustments were made by several observers according to the instructions given above. It was found that while there was considerable variation among the various adjustments made, the average of all such adjustments on a lamp was very close to the point of maximum efficiency.

From a study of the pressure-efficiency curves (Fig. 4), it will be noted that this slight decrease in the gas consumption may have still another helpful effect. This is true since such a change in adjustment will assist in correcting the effects of changes in gas pressure in cases where the pressure at which the lamp is operated is higher than the pressure at time of adjustment, a condition not infrequently found especially in cities where many lamps are periodically visited by the gas company or lamp companies for cleaning or adjustment.

5. KIND OF GAS

In making comparisons of the operation of a lamp with various gases all conditions other than the quality of the gas supply and the adjustment of the lamp were kept as constant as possible. In order to facilitate this, comparison was made only between results obtained on the same day and using the same mantle, glassware, and measuring apparatus. Observations of barometric pressure, humidity, and temperature were made with sufficient frequency so that any marked change in conditions would be noticed and the proper corrections applied. In comparing two gases the pressure at the burner was kept constant throughout the test, but the adjustments of the gas supply and the air shutter were altered to ascertain the most favorable condition for each gas. A set of photometric readings was taken at each adjustment and the rate of gas consumption was determined. From these data the efficiency of the lamp in lumens per Btu per hour was calculated. From the values obtained the candlepower and efficiency curves were plotted in the typical form. (See Fig. 7.) From these curves for each run the candlepower, efficiency, and consumption of gas could be noted for the points of maximum

candlepower and of maximum efficiency. Comparisons of gases were made between the observations of a single day at these two points.

Such comparisons were made between lean water gas and the city supply of rich mixed gas, between lean water gas and lean coal gas, and between the city supply and a specially enriched gas prepared by introducing benzol vapor into the city gas (changing the gas from 15 candles and 630 Btu to 21 candles and 650 Btu or

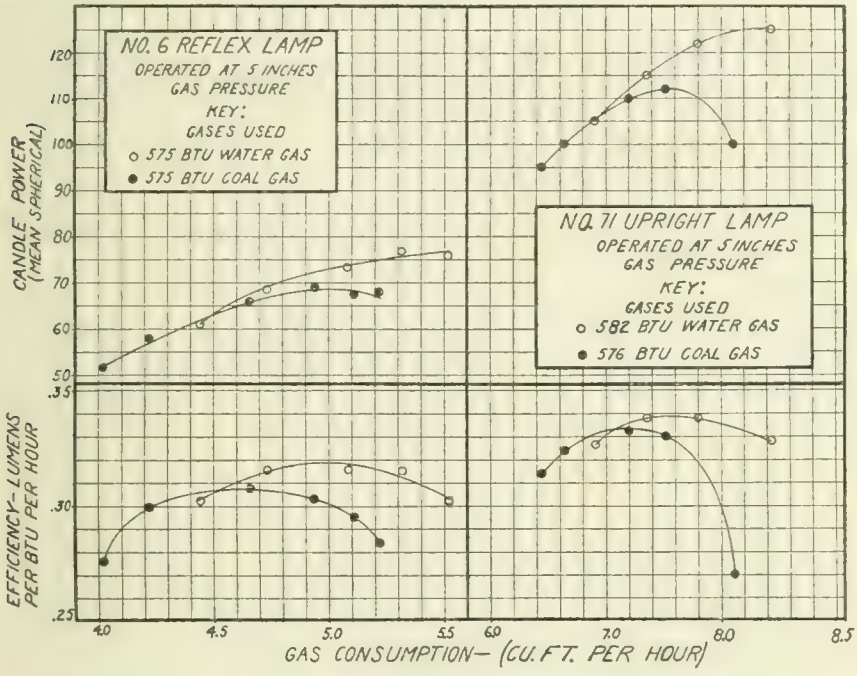

NO. 6 REFLEX LAMP
OPERATED AT 5 INCHES GAS PRESSURE
KEY:
GASES USED
○ 575 BTU WATER GAS
● 575 BTU COAL GAS
NO. 11 UPRIGHT LAMP
OPERATED AT 5 INCHES GAS PRESSURE
KEY:
GASES USED
○ 582 BTU WATER GAS
● 576 BTU COAL GAS
CANDLE POWER (MEAN SPHERICAL)
EFFICIENCY—LUMENS PER BTU PER HOUR
GAS CONSUMPTION—(CU. FT. PER HOUR)

FIG. 7.—*Comparative operation of lamps with coal gas and water gas of about the same total heating value*

higher). The nature of these gases can be seen by consulting the summary of analyses in the appendix to this paper. (See p. 48.)

The comparison of lean water gas and the city supply showed the influence of the differences in total heating value for gases of rather similar composition. The benzolized gas was used to ascertain the influence of this hydrocarbon vapor upon the efficiency of the lamp. The influence of the different proportions of hydrogen, carbon monoxide, and methane, in coal gas as compared with water gas, is shown by the third series of comparisons.

Table 5 presents the results of these comparisons between the lean water gases and the city gas which was largely water gas.

Only the data for the points of maximum candlepower and of maximum efficiency are presented, since comparisons are feasible only under these two conditions. The results for candlepower consumption of gas, and efficiency are taken from curves like those of Fig. 7. The efficiency change per 100 Btu decrease in heating value per cubic foot is computed by dividing 100 times the difference in efficiency of the two gases of each pair by the difference in the heating value of the gases. When the value is plus, it indicates an advantage in luminous efficiency for the leaner gas, when minus, a disadvantage. In considering these data and those of the two tables following, comparison should be made only between the two tests of each pair; comparison between the results for different lamps should not be made except in a very general way.

TABLE 5.—Influence of Heating Value of Water Gas Upon Efficiency in Use

UPRIGHT LAMPS

Test No.	Pressure of gas, inches.	Gas No.	Heating value of gas, Btu per cubic foot	Adjusted to maximum candlepower					Adjusted to maximum efficiency				
				Mean spherical candlepower	Gas consumption		Efficiency lumens per Btu per hour	Change in lumens per 100 Btu	Mean spherical candlepower	Gas consumption		Efficiency lumens per Btu per hour	Change in lumens per 100 Btu
					Cubic feet per hour	Btu per hour				Cubic feet per hour	Btu per hour		
1	3	104	686	81	5.7	3910	0.255	.....	78	5.4	3700	0.265	.....
		105	570	93	7.1	4050	.290	+0.03	92	6.8	3880	.295	+0.03
2	3	103	629	81	5.9	3710	.275	.....	80	5.7	3580	.285	.....
		102	573	96	7.2	4070	.295	+ .04	95	7.0	4010	.300	+ .03
c3	3	104	686	69	4.3	2950	.295	.....	68	4.2	2880	.305	.....
		105	570	72	5.1	2910	.315	+ .02	70	4.8	2740	.320	+ .01
c4	3	103	629	71	6.2	3900	.220	.....	70	6.0	3770	.235	.....
		102	573	74	7.0	4010	.230	+ 02	74	6.6	3780	.240	+ .01
c5	3	103	629	103	6.1	3840	335	.....	96	5.7	3580	.340	.....
		102	573	b 104	7.2	4130	b 315	− .04	99	6.3	3610	.340	.00
c6	2	12	663	78	6.5	4310	.230	.....	74	5.8	3840	.245	.....
		15	548	80	7.8	4270	.265	+ .03	80	7.5	4110	.275	+ .02
c7	2	63	648	73	5.6	3630	.260	.....	68	4.8	3110	.275	.....
		77	548	b 60	5.6	3070	b. 285	+ .03	b 69	5.6	3070	b. 285	+ .01
c8	3	14	670	94	6.5	4360	.245	.....	86	5.7	3820	.285	.....
		15	548	109	7.8	4270	.255	+ .01	98	7.5	4110	.280	.00
c9	3	63	648	90	5.7	3690	.310	.....	87	5.4	3500	.315	.....
		77	548	b 96	6.8	3730	b. 325	+ .02	95	6.7	3670	.325	+ .01
c10	5	.....	656	111	8.5	5660	.245	.....	102	6.6	4390	.290	.....
		15	548	132	10.1	5540	.300	+ .05	129	9.5	5200	.310	+ .02
c11	5	74	631	113	7.8	4920	.290	.....	106	6.8	4290	.310	.....
		73	550	127	9.5	5220	.305	+ .02	121	8.7	4790	.315	+ .01
Maximum values				132	10.1	5660	.335	+ .05	129	9.5	5200	.340	+ .03
Minimum values				69	4.3	2910	.220	− .04	68	4.2	2740	.235	.00
Average				87	6.4	4100	.275	+ .02	88	6.3	3792	.292	+ .01

a Cheaper grades of lamps used.

b This value was the maximum obtainable under the conditions of the experiment, but the value does not represent a true maximum.

c Readings made in sphere.

TABLE 5.—Influence of Heating Value of Water Gas Upon Efficiency in Use— Continued

JUNIOR LAMPS

Test No.	Pressure of gas, inches.	Gas No.	Heating value of gas, Btu per cubic foot	Adjusted to maximum candlepower: Mean spherical candlepower	Gas consumption: Cubic feet per hour	Gas consumption: Btu per hour	Efficiency lumens per Btu per hour	Change in lumens per 100 Btu	Adjusted to maximum efficiency: Mean spherical candlepower	Gas consumption: Cubic feet per hour	Gas consumption: Btu per hour	Efficiency lumens per Btu per hour	Change in lumens per 100 Btu
12	3	106	650	45	2.4	1560	0.355	.....	43	2.3	1490	0.365	.....
		107	554	46	2.9	1610	.355	0.00	45	2.7	1500	.360	−0.01
[a] 13	3	106	651	37	2.2	1430	.320	.....	35	2.0	1300	.340	.....
		107	554	40	2.6	1440	.345	+ .03	37	2.4	1330	.355	+ .02
[a] 14	3	106	650	38	2.2	1430	.340	.....	38	2.1	1360	.350	.....
		107	554	42	2.9	1610	.330	− .01	41	2.6	1440	.360	+ .01
[b] 15	2	[a] 77	680	39	2.2	1500	.330	.....	37	2.0	1360	.345	.....
		77	548	38	2.5	1370	.380	+ .04	37	2.5	1370	.385	+ .03
[b] 16	3	.....	647	41	2.4	1550	.320	.....	38	2.2	1420	.330	.....
		21	530	38	3.0	1590	.310	− .01	37	2.9	1540	.315	− .01
[b] 17	3	[a] 75	636	45	2.6	1650	.330	.....	43	2.4	1530	.365	.....
		77	548	47	2.5	1370	.345	+ .02	46	2.4	1320	.365	.00
[b] 18	5	75	624	49	3.1	1930	.325	.....	44	2.6	1620	.345	.....
		77	548	51	3.7	2030	.325	.00	48	3.3	1810	.335	− .01
Maximum values				51	3.7	2030	.360	+ .03	48	3.3	1810	.385	+ .05
Minimum vaues				37	2.2	1370	.310	− .01	35	2.0	1300	.330	− .01
Average				31	2.7	1580	.336	+ .01	41	2.5	1460	.355	+ .00

C. E-Z LAMPS

21	3	98	646	62	3.2	2070	0.390	+0.02	62	3.0	1940	0.405	.....
		97	537	69	3.9	2090	.410	.....	66	3.7	1990	.420	+0.01
22	3	98	646	63	2.8	1810	.425	+ .03	62	2.6	1680	.455	.....
		97	537	76	4.0	2150	.455	.....	73	3.6	1930	.485	+ .03
23	3	98	646	65	3.1	2000	.415	.00	61	2.9	1870	.435	− .01
		97	537	72	4.0	2150	.415	.....	67	3.7	1990	.420	.....
[b] 24	2	24	640	52	3.1	1980	.340	.....	49	2.7	1730	.355	− .01
		23	544	58	4.0	2170	.335	− .01	55	3.6	1960	.350	.....
[b] 25	2	64	618	51	2.9	1790	.365	− .01	49	2.6	1610	.390	− .02
		81	544	60	3.9	2120	.360	.....	55	3.4	1850	.375	.....
[b] 26	3	24	640	60	3.4	2180	.345	.00	54	2.9	1860	.375	− .02
		23	544	59	4.0	2180	.345	.....	53	3.5	1900	.355	.....
[b] 27	3	64	618	64	3.4	2100	.380	− .04	56	2.9	1790	.385	.00
		81	544	67	4.5	2450	.350	.....	61	3.7	2010	.385	.....
[b] 28	5	26	652	69	3.9	2540	.340	− .01	64	3.2	2090	.385	− .01
		23	544	77	5.5	2990	.325	.....	70	4.4	2390	.370	.....
[b] 29	5	72	631	73	4.1	2590	.355	− .01	62	3.2	2020	.385	+ .01
		73	550	80	5.2	2860	.345	.....	70	4.0	2200	.395	.....
Maximum value				80	5.5	2990	.455	+ .03	70	4.4	2390	.485	+ .03
Minimum value				51	2.3	1790	.325	− .04	49	2.6	1610	.350	− .02
Average				66	3.8	2234	.372	.00	[illegible]	3.3	1989	.395	.00

[a] Cheaper grades of lamps used. [b] Readings made in sphere.

TABLE 5.—Influence of Heating Value of Water Gas Upon Efficiency in Use—Continued

REFLEX AND INVERTED LAMPS

Test No.	Pressure of gas, inches.	Gas No.	Heating value of gas, Btu per cubic foot	Adjusted to maximum candlepower: Mean spherical candlepower	Gas consumption: Cubic feet per hour	Gas consumption: Btu per hour	Efficiency lumens per Btu per hour	Change in lumens per 100 Btu	Adjusted to maximum efficiency: Mean spherical candlepower	Gas consumption: Cubic feet per hour	Gas consumption: Btu per hour	Efficiency lumens per Btu per hour	Change in lumens per 100 Btu
30	3	91	663	87	4.0	2650	0.420	−0.02	84	3.7	2450	0.425	−0.02
		92	580	87	4.6	2670	.405	.....	87	4.6	2670	.405	.....
31	3	98	646	a 86	4.5	2900	.370	.00	80	4.0	2580	.390	− .09
		97	537	74	4.6	2470	.375	.....	a 74	4.6	2470	a .380	.....
32	3		650	82	4.0	2620	.385	− .04	79	3.9	2540	.400	− .09
		87	566	70	4.4	2490	.350	.....	67	4.3	2430	.380	.....
33	3	91	663	a 81	3.8	2520	.405	− .07	80	3.7	2550	.410	− .05
		92	580	a 70	4.3	2490	.365	.....	a 70	4.3	2490	a .365	.....
34	3	90	654	77	4.4	2880	.335	− .04	69	3.8	2480	.365	+ .05
		87	566	a 77	4.8	2720	.300	.....	72	4.4	2490	.370	.....
35	3	91	663	77	4.1	2720	.355	− .01	71	3.6	2390	.370	− .01
		92	580	79	5.0	2900	.345	.....	74	4.5	2610	.360	.....
36	3	91	663	a 87	4.8	3180	.335	+ .06	80	4.5	2980	.345	+ .04
		93	574	85	4.8	2760	.385	.....	81	4.6	2640	.385	.....
37	5	101	658	87	5.0	3290	.330	+ .03	79	4.1	2700	.385	− .01
		100	567	90	5.8	3290	.355	.....	84	5.0	2830	.375	.....
38	5	106	651	89	4.8	3120	.360	+ .02	75	4.3	2800	.375	+ .07
		107	554	94	5.6	3100	.380	.....	89	5.0	2770	.400	.....
39	5	108	650	95	4.9	3190	.375	+ .03	88	4.1	2660	.405	+ .02
		109	561	98	5.4	3030	.405	.....	93	5.0	2810	.420	.....
40	3	98	646	64	3.7	2390	.335	− .02	61	3.4	2200	.340	− .01
		97	537	61	4.6	2470	.310	.....	57	4.1	2200	.325	.....
41	5	101	658	66	4.2	2760	.300	.00	61	3.5	2300	.335	− .01
		100	567	66	5.0	2830	.300	.....	61	4.2	2380	.325	.....
42	2	68	640	a 72	3.9	2490	.370	− .03	70	3.8	2430	.370	− .03
		66	553	a 58	3.8	2100	.345	.....	a 58	3.8	2100	a .345	.....
43	3	18	651	71	4.6	2990	.305	+ .03	65	3.8	2470	.335	.00
		19	537	a 71	4.9	2630	.335	.....	69	4.8	2580	.335	.....
44	3	68	640	81	4.7	3010	.375	− .01	74	3.9	2490	.370	+ .01
		66	553	78	4.8	2650	.370	.....	75	4.5	2490	.380	.....
45	5	18	651	78	5.0	3250	.300	+ .01	72	4.2	2730	.335	.00
		19	537	86	6.4	3440	.315	.....	77	5.3	2840	.340	.....
46	5	68	640	87	5.0	3190	.340	+ .01	81	4.3	2750	.370	+ .01
		69	550	99	6.0	3300	.345	.....	85	5.1	2800	.380	.....
47	1	80	641	a 43	3.7	2370	.225	+ .07	40	3.3	2120	.215	+ .05
		79	567	53	4.3	2440	.275	.....	51	4.0	2270	.285	.....
48	3	71	636	a 62	3.9	2480	.310	− .02	a 62	3.9	2480	.310	.00
		69	590	70	5.0	2950	.300	.....	66	4.6	2720	.310	.....
Maximum value				98	6.4	3440	.420	+ .07	93	5.3	2980	.425	+ .07
Minimum value				43	3.7	2100	.225	− .04	40	3.3	2100	.215	− .09
Average				74	4.7	2810	.344	.00	73	4.4	2565	.364	.00

a The value is the maximum obtainable under the conditions of the experiment, but the value does not represent a true maximum.

b Omission in reading of lamps used.

c Made on [illegible]

Some of the comparisons, as indicated in footnotes, were made in the integrating sphere; the other tests were made with the simple photometer at the appropriate angle, but are computed to show the mean spherical candlepower corrected for variations in humidity to a standard condition of 8 liters of water vapor per cubic meter of air (equivalent to 0.8 per cent water vapor by volume).

TABLE 6.—Comparison of Efficiencies of City Gas and Benzolized Gas

REFLEX LAMPS

Test No.	Pressure of gas, inches	Gas No.	Heating value of gas, Btu per cubic foot	Adjusted to maximum candlepower					Adjusted to maximum efficiency				
				Mean spherical candlepower	Gas consumption		Efficiency lumens per Btu per hour	Change in lumens per 100 Btu	Mean spherical candlepower	Gas consumption		Efficiency lumens per Btu per hour	Change in lumens per 100 Btu
					Cubic feet per hour	Btu per hour				Cubic feet per hour	Btu per hour		
[a] 49	5	121	[b] B 651	84	4.3	2800	0.375	.....	83	4.2	2730	0.375	.....
		122	631	84	4.8	3030	.345	−0.15	79	4.3	2720	.370	−0.03
51	3	111	B 726	66	3.5	2540	.330	.....	62	3.3	2390	.335	.....
		111	670	70	3.8	2550	.345	+ .03	64	3.4	2280	.355	+ .04

UPRIGHT LAMPS

[a] 50	3	121	B 651	86	5.5	3580	0.300	.....	83	5.2	3380	0.310	.....
		123	633	85	5.8	3670	.295	−0.03	82	5.3	3290	.305	−0.03
[a] 52	5	121	B 651	107	6.5	4230	.315	.....	98	5.8	3780	.330	.....
		123	633	110	6.5	4110	.325	+ .06	106	6.1	3860	.330	.00
53	2	115	B 737	[illegible]	5.4	3980	.265	.....	80	4.8	3540	.285	.....
		116	663	82	6.5	4310	.240	− .03	79	5.7	3780	.255	− .04
54	3	113	B 724	100	6.9	5000	.290	.....	90	5.0	3520	.310	.....
		113	670	99	6.5	4360	.285	− .01	90	5.1	3420	.300	− .02
55	5	.....	B 724	112	6.8	4920	.310	.....	112	5.9	4270	.330	.....
		114	670	118	8.5	5700	.260	− .09	108	6.6	4420	.310	− .04
56	5	115	B 737	113	6.8	5010	.280	.....	108	5.9	4350	.310	.....
		116	663	114	8.6	5700	.250	− .04	106	6.6	4370	.305	− .01

[a] These runs are of doubtful significance on account of the small differences in the heating values of the gases compared; therefore results in these tables have not been averaged.

[b] B = Benzolized gas.

The use of cheaper grades of lamps or of lamps of different construction than those employed in most of the comparisons is indicated in the footnotes.

Where the curves did not reach a true maximum of candlepower or efficiency within the range of consumption which could be obtained with the pressure in use, this fact is noted in the table. The value given in such cases represents the maximum attained. In these cases the comparisons of efficiency are, of course, some-

what less significant than otherwise. The reason that maximum candlepower or efficiency was not reached is believed to have been small amounts of dust in the gas orifice; but in a few cases it was the result of low pressure (tests 7 and 42) or of peculiarities of the individual lamps used (tests 5, 47, and 48).

TABLE 7.—Comparison of Efficiencies of Coal Gas and Water Gas of Approximately Equal Total Heating Value

REFLEX LAMPS

Test No.	Pressure of gas, inches	Gas No.	Heating value of gas, Btu per cubic foot	Adjusted to maximum candlepower: Mean spherical candlepower	Gas consumption: Cubic feet per hour	Gas consumption: Btu per hour	Efficiency lumens per Btu per hour	Efficiency change	Adjusted to maximum efficiency: Mean spherical candlepower	Gas consumption: Cubic feet per hour	Gas consumption: Btu per hour	Efficiency lumens per Btu per hour	Efficiency change
								Per ct.					Per ct.
57	3	117	[a]C 576	59	4.5	2590	0.280	.....	58	4.4	2450	0.285	.....
		118	[b]W 575	[c]56	4.4	2530	.280	0	[c]56	4.4	2530	.280	1.7
58	5	117	C 576	72	5.0	2870	.315	.....	68	4.6	2650	.320	.....
		118	W 575	79	5.4	3100	.325	3.2	76	5.0	2870	.335	4.7
59	5	120	C 582	69	4.9	2850	.305	.....	66	4.6	2680	.315	.....
		118	W 568	[c]76	5.0	2840	.305	0	65	4.5	2560	.320	1.6
Maximum value				79	5.4	3100	.325	3.2	76	5.0	2870	.335	4.7
Minimum value				56	4.4	2530	.280	0	56	4.4	2450	.280	1.6
Average				68	4.9	2800	.300	1.0	65	4.6	2620	.310	2.7

UPRIGHT LAMPS

60	3	117	C 576	91	6.1	3510	0.310	.....	87	5.3	3050	0.320	.....
		116	W 582	98	6.7	3900	.315	1.6	94	6.3	3670	.325	1.5
61	5	117	C 576	107	7.5	4320	.315	.....	106	7.2	4140	.320	.....
		116	W 582	123	8.4	4890	.315	0	116	7.6	4420	.330	3.1
Maximum value				123	8.4	4890	.315	1.6	116	7.6	4420	.330	3.1
Minimum value				91	6.1	3510	.310	0	87	5.3	3050	.320	1.5
Average				105	7.2	4150	.315	.8	101	6.6	3870	.325	2.3

[a] C = coal gas.

[b] W = water gas.

[c] This value was the maximum obtainable under the conditions of the experiment, but the value does not represent a true maximum.

Table 6 gives corresponding data for comparisons between city gas and benzolized gas; and Table 7 a comparison between coal gas and water gas of about the same total heating values. However, in Table 7 the differences in efficiency shown are absolute differences; that is, are not computed per 100 Btu change in heating value. The differences marked plus represent an advantage for water gas; those marked minus, an advantage for coal gas.

Some measurements with natural gas from southern Ohio, which were made shortly before this investigation was begun, are also included here as Table 8. The total heating value of the natural gas was 1120 Btu per cubic foot; its composition is given in the appendix. It was compared with a mixed gas of about 625 Btu per cubic foot.

TABLE 8.—Comparison of Natural Gas with Manufactured City Gas

[Heating Values, 1120 and 625 Btu per cubic foot. Pressure, 7 and 3 Inches of Water.]

Pressure of gas, inches	Heating value of gas, Btu per cubic foot	Adjusted to maximum candlepower					Adjusted to maximum efficiency				
		Mean spherical candlepower	Gas consumption		Efficiency lumens per Btu per hour	Per cent efficiency	Mean spherical candlepower	Gas consumption		Efficiency lumens per Btu per hour	Change in lumens per 100 Btu
			Cubic feet per hour	Btu per hour				Cubic feet per hour	Btu per hour		
REFLEX LAMPS											
7	[a]N 1120	63	2.4	269	0.295	0.014	61	2.3	258	0.300	0.017
3	625	80	4.7	294	.340	.....	67	4.3	269	.360	.....
UPRIGHT LAMPS											
7	N 1120	65	3.2	358	0.230	0.021	64	2.9	325	0.250	0.023
3	625	68	6.0	375	.295	.....	80	5.0	313	.320	.....
JUNIOR LAMPS											
7	N 1120	24	1.2	135	0.225	0.025	23	1.1	123	0.230	0.030
3	625	35	2.4	150	.295	.....	35	2.1	131	.330	.....
C. E-Z LAMPS											
7	N 1120	46	2.0	224	0.260	0.020	40	1.5	168	0.300	0.015
3	625	56	3.5	219	.320	.....	49	2.8	175	.350	.....

[a] N=Natural gas.

Between the natural gas and manufactured gas comparisons can not be made with conditions so closely similar as is done in comparing two kinds of gas which are more nearly alike. For example, instead of burning the two gases in succession in the same lamp, each gas must be burned in a lamp adapted to it. Moreover, the pressures used for tests, being like those commercially prevailing, are decidedly different for the two types of gas. In these tests, the gas was used at pressures which were considered to represent common practice; these were 7 inches of water (approximately 4 ounces per square inch) for the natural gas and 3 inches for the manufactured gas.

The data given in most cases represent measurements on a single lamp of each kind, but some of the results for manufactured gas are the averages for two or three lamps of measurements which were made within a few days of the time when the natural gas was tested. Exact conclusions can not be drawn from so small a number of measurements, but the degree of consistency of the results seems to justify the statement that in regular commercial lamps natural gas may be expected to give an efficiency (per heat unit) about 20 per cent lower than manufactured gas.

To summarize all the data on manufactured gas in a convenient form Tables 9 and 10 have been prepared, showing for the four styles of lamp the changes in efficiency expressed as percentages of the average efficiency. The following general conclusions are drawn from these data:

1. The upright lamps show a distinctly higher efficiency with the lean water gas than with the city gas, both at maximum-candlepower and maximum-efficiency adjustments; and the advantage is greater at maximum candlepower than at maximum efficiency.

2. The Junior lamps show a distinctly higher efficiency at maximum-candlepower adjustments with the lean gas than with the city gas; but at maximum-efficiency adjustments there is practically no difference between the efficiencies obtained.

TABLE 9.—Lean Water Gas versus City Gas

[Percentage increase in efficiency with 100 Btu per cubic foot decrease in heating value]

Lamp	Number of comparisons	Maximum-candlepower adjustment	Maximum-efficiency adjustment
		Per cent	Per cent
Upright	11	7	3
Inverted	19	0	0
Junior	7	3	0
C. E-Z	9	0	0

3. The inverted and C. E-Z lamps show no difference in efficiency, within the limits of experimental uncertainties, with the lean water gas and the city gas either at maximum-efficiency or maximum-candlepower adjustments.

4. The upright-mantle lamps show, so far as the tests are significant, a distinct advantage of the benzol-enriched gas, both at maximum-candlepower and maximum-efficiency adjustments. As will be seen from Table 6, an insufficient number of runs was

made to show conclusively whether the presence of considerable benzol in the city gas had any marked effect on the efficiency of the inverted-mantle type of lamps. On account of the small difference between the heating values of the gases used in test 49, the difference found is probably not significant. Test 51 shows an advantage of the leaner gas, but more tests would be necessary to confirm this.

5. With both upright and inverted mantle lamps at either maximum candlepower or maximum efficiency there is a distinct, but small, advantage of water gas over coal gas of the same total heating value, except in some cases at very low pressures (less than 2 inches), when lean water gas may not show as high efficiencies as rich water gas or lean coal gas on account of the inability of the lamp to pass sufficient of the lean water gas to attain a true maximum efficiency. This advantage of water gas over coal gas probably results largely from the smaller loss of heat in the products with water gas than with coal gas of the same total heating value. (The lower net heating value is also a result of this.) However, there are some differences in flame characteristics that result from difference in the proportions of hydrogen, carbon monoxide, and hydrocarbons, and these are perhaps fully as important as the differences in heat losses.[11] In this connection it is interesting to note that we have found a distinct indication of higher heat input at maximum efficiency or at maximum candlepower with water gas than with coal gas except in those tests where the true maximum adjustment was not reached. (See especially Fig. 7.)

TABLE 10.—Advantage of Lean Water Gas over Lean Coal Gas of Approximately Equal Total Heating Value

[Percentages refer to difference in efficiency, i. e., differences in lumens per Btu per hour in favor of the water gas]

Lamp	Number of comparisons	Maximum-candlepower adjustment	Maximum-efficiency adjustment
		Per cent	Per cent
Upright	2	1	2
Inverted	3	1	3

Greater ease of adjustment with lean gas than with rich gas is often claimed, but we have not been able to prove conclusively any such influence. There are distinct indications that the range

[11] The influcnee of these flame characteristics is being investigated further at this Bureau.

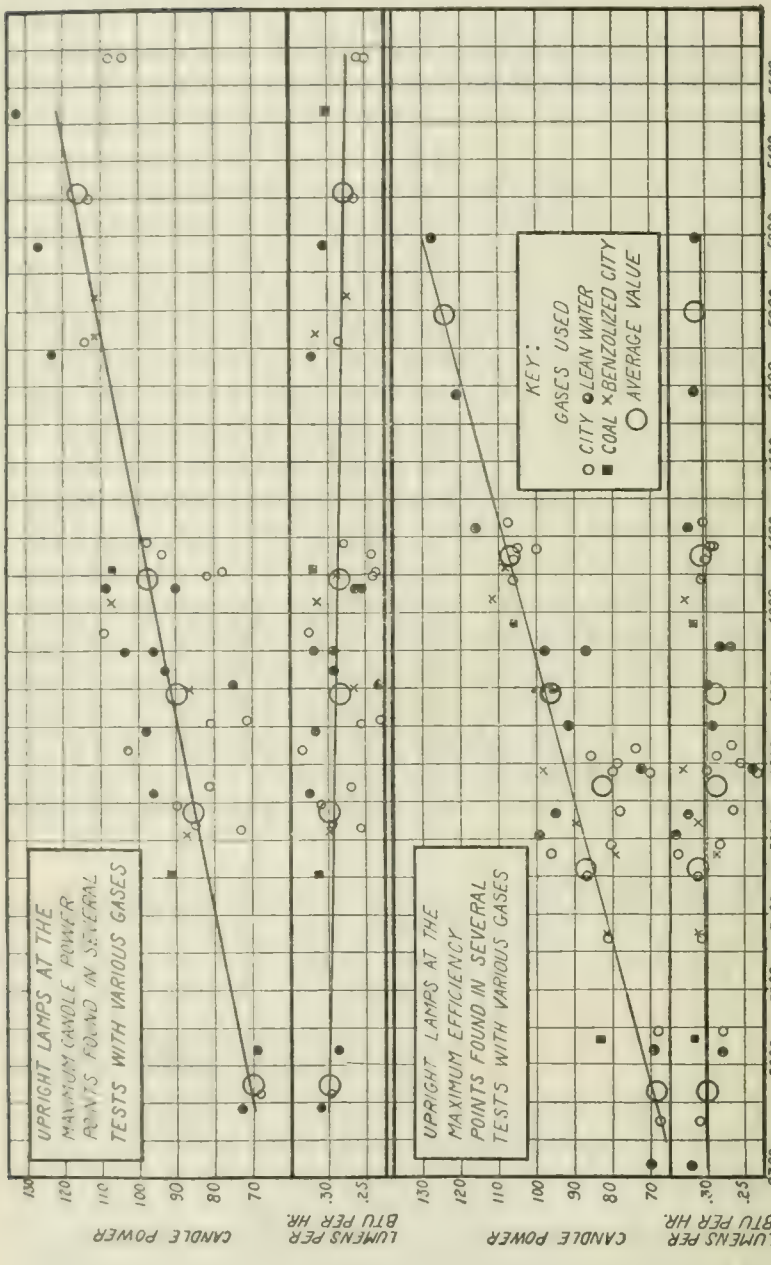

UPRIGHT LAMPS AT THE MAXIMUM CANDLE POWER POINTS FOUND IN SEVERAL TESTS WITH VARIOUS GASES
UPRIGHT LAMPS AT THE MAXIMUM EFFICIENCY POINTS FOUND IN SEVERAL TESTS WITH VARIOUS GASES
KEY:
GASES USED
○ CITY ● LEAN WATER
■ COAL × BENZOLIZED CITY
◯ AVERAGE VALUE
CANDLE POWER
LUMENS PER BTU PER HR.
HEAT INPUT — (BTU PER HOUR)

FIG. 8.—*Performance of various upright-mantle lamps with various gases at various rates of heat input*

of air-shutter adjustment is wider with lean gas (see preceding section) and somewhat less definite indication that near the point of maximum efficiency there is a wider range of gas rate for equally good efficiency. Beyond this point, however, we would not be justified in drawing any conclusion as to the advantages of leaner

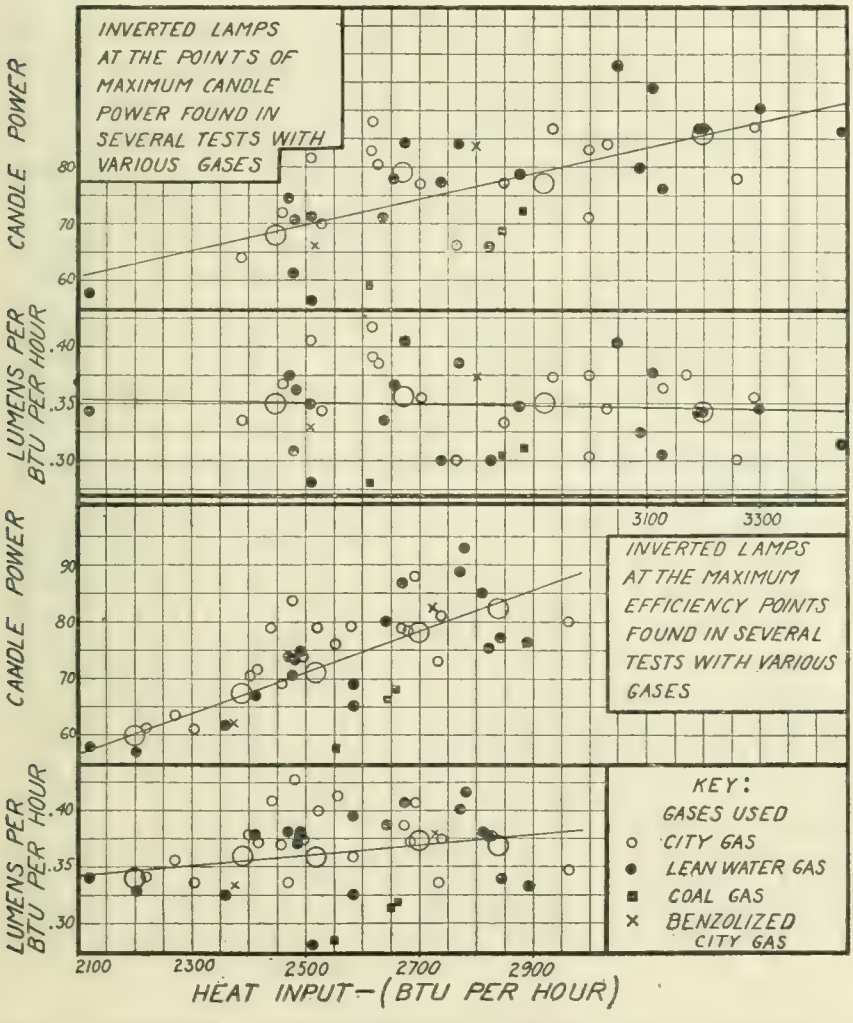


FIG. 9.—*Performance of inverted-mantle lamps with various gases at various rates of heat input*

gas. It was also noted that when operated on lean water gas with wide-open air shutters, the inverted-mantle and C. E-Z lamps become noisy at very low pressures and in a few cases back-fire. No noticeably greater tendency to carboning of mantles was observed with one quality of gas than with another, provided the

gas passages of the lamp were clear and the air shutters were wide open. This subject is discussed at greater length in a report on field inspections of mantles, which includes a summary of carboned mantles as evidence of this point.[13]

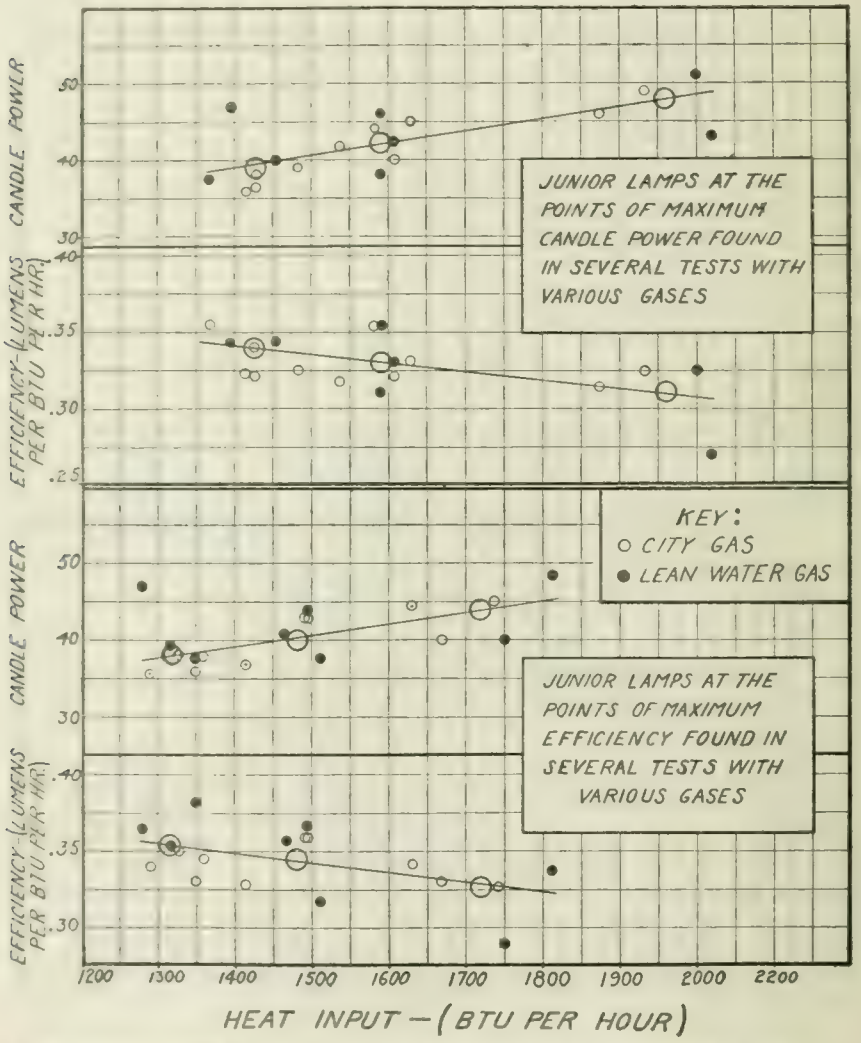


FIG. 10.—*Performance of Junior lamps with various gases at various rates of heat input*

A further study of the relation of candlepower and efficiency to the rate of heat input under conditions of maximum candlepower and maximum efficiency is shown in the curves of Figs. 8–11, which are plotted from the data of Tables 5–7, to show all

[13] Bureau of Standards, Technologic Paper No. 99, by R. S. McBride and C. E. Reinicker.

the maximum candlepower and efficiency values for all the gases tested. These values are from several lamps operated under various pressures and other conditions. The curves are so proportioned that the same percentage change in heat input is of practically the same apparent magnitude in the several figures.

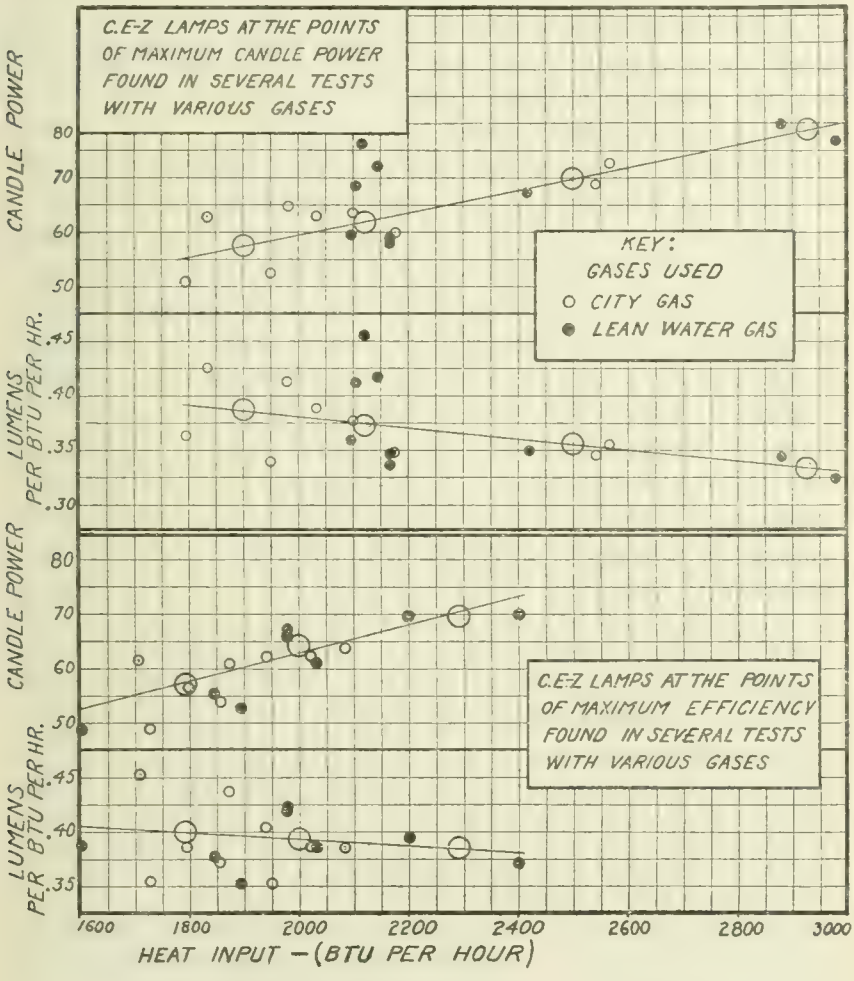

C.E-Z LAMPS AT THE POINTS OF MAXIMUM CANDLE POWER FOUND IN SEVERAL TESTS WITH VARIOUS GASES
CANDLE POWER
LUMENS PER BTU PER HR.
KEY:
GASES USED
CITY GAS
LEAN WATER GAS
C.E-Z LAMPS AT THE POINTS OF MAXIMUM EFFICIENCY FOUND IN SEVERAL TESTS WITH VARIOUS GASES
HEAT INPUT — (BTU PER HOUR)

FIG. 11.—*Performance of C. E-Z lamps with various gases at various rates of heat input*

From these curves it will be seen that the higher the heat input of a lamp happens to be at maximum candlepower or at maximum efficiency, the higher is the candlepower produced. This is to be expected unless there was a marked decrease in efficiency at the higher rates.

If adjusted from time to time to maintain maximum efficiency under changing atmospheric or other conditions, the smaller lamps seem to show markedly lower efficiencies the higher the heat input per hour for such adjustment happens to be. With similar variations the upright lamps do not change in efficiency, while the inverted lamps show higher efficiencies with higher rates of heat input. When the adjustment to maximum candlepower is maintained, the small lamps and the upright lamps show behaviors similar to those just described, but the inverted lamps show lower efficiencies with higher rates of heat input.

If very exact determination of the relation between heat input and efficiency were possible, it is presumed that there would be found some optimum rate of heat input for each lamp with each gas. The degree of reproducibility attainable in the adjustment and condition of gas lamps is, however, not sufficient to permit determination of this optimum point and hence the curves of efficiency given in Figs. 8–11 are drawn as straight lines as nearly as possible through the centers of gravity (shown as ⊙ on the curves) of the various groups of individual entries. The degree of uncertainty in position or slope of these curves is evident from a consideration of the scattering of individual points on the plots.

The variation in candlepower and efficiency of the lamps at the points of maximum candlepower and maximum efficiency is the result of the large number of variables which affect these results, including lamp conditions, atmospheric conditions, and variations in the gas itself. But even considering all of these variables it is rather surprising that for these adjustments of maximum candlepower and maximum efficiency there should be found such great differences in the rate of heat input as is shown by these curves; however, it was not found that the heating value or kind of gas used had any consistent effect upon the rate of heat input at these maximum points.

This variation shows how difficult it is to secure any significant photometric results on gas-mantle lamps without extremely long series of comparisons; and it makes clear why the calculations of various experimenters often disagree because of the limited number of tests used by each as a basis of his conclusion.

If an experimenter is to secure reproducible results, he must have perfect control of the atmospheric conditions, including barometric pressure, humidity, and freedom from dust, absolutely clean passages in the lamp for gas and air, uniformity of mantle composition and quality, freedom from drafts, and a very unusual

mechanical perfection in the form of lamp, since even a very slight irregularity or lack of symmetry of the entering gas stream will often have a very marked effect on the amount of air entrained and on the position of the flame in the mantle, thus affecting both the total output and the distribution of the light. This high mechanical perfection may be of little consequence in the ordinary use of the lamp; but for accuracy of laboratory tests it is an essential feature.

6. FLUCTUATION OF GAS QUALITY

A series of tests was undertaken to ascertain the effect upon the efficiency of a lamp of changing the quality of the gas supplied to it without altering the adjustments of the gas and air supply. The set-up for these tests was so arranged that either of two different gases or a mixture of them in any desired proportions could be supplied to the lamp. Each gas was metered separately and when a mixture was used the mixture was also metered by a third meter. When a mixture of gases was used, the heating value of the mixture was computed from the proportion of gases of known heating value present.

In performing a test, the lamp being tested was adjusted to produce its maximum candlepower when operated at a fixed pressure with a given gas of known heating value. The meter passing the mixed gas was allowed to operate a sufficient length of time on the determined mixture, to purge out any other gas remaining in it. A set of photometric observations was made and the rate of gas consumption noted. From these data the efficiency of the lamp under the existing conditions could be computed. The gas quality supplied to the lamp was then changed, the adjustment of the lamp remaining unchanged. Observations were made as before and the efficiency again computed. The lamp having been operated on various gases at one adjustment, the adjustment was then changed to produce maximum candlepower with a different gas or mixture of gases. The efficiency of operation was then determined with different gases under the new condition.

The results obtained in typical series for four kinds of lamps are shown by the curves of Fig. 12. In the work there shown the gases used were water gas from 540 to 575 Btu, city gas from 625 to 660 Btu, and gas specially enriched with benzene vapor to about 750 Btu.

As will be noted from these curves, there is in most cases a distinct increase in operating efficiency when a lamp adjusted to maximum candlepower with one gas is operated on a leaner gas without change of adjustment, and a more marked decrease

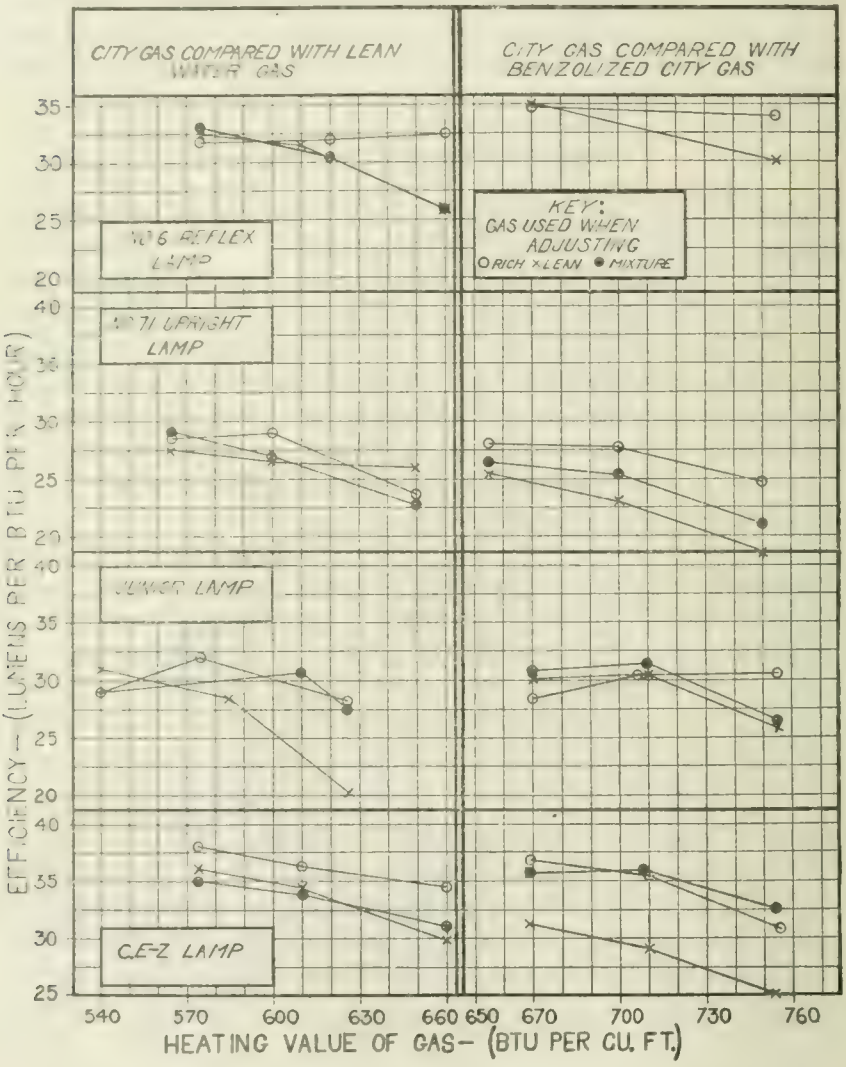

CITY GAS COMPARED WITH LEAN WATER GAS
CITY GAS COMPARED WITH BENZOLIZED CITY GAS
KEY:
GAS USED WHEN ADJUSTING
O RICH × LEAN ● MIXTURE
No 6 REFLEX LAMP
No 7 UPRIGHT LAMP
JUNIOR LAMP
C.E-Z LAMP
EFFICIENCY — (LUMENS PER BTU PER HOUR)
HEATING VALUE OF GAS — (BTU PER CU. FT.)
540 570 600 630 660 650 670 700 730 760

FIG. 12.—*Influence of gas quality on lamp efficiency with fixed adjustment*

in efficiency when a lamp adjusted with lean gas is operated with richer gas, all other conditions remaining unchanged. The explanation of this seems to be that when a lamp is adjusted to produce maximum candlepower with a given gas it is using more

gas than when adjusted to maximum efficiency, except in unusual cases when the maximum-candlepower and maximum-efficiency points are coincident. Consequently, a lamp adjusted to produce maximum candlepower with a rich gas is brought nearer to its maximum-efficiency adjustment when a lower quality gas is substituted, the diminution in the heat supplied in the latter case tending to bring the lamp nearer to this maximum-efficiency point. On the other hand, if a lamp adjusted to give maximum candlepower on a lean gas is operated without readjustment on a rich gas, the result is that it is supplied with such an excess of heat over the amount which it can efficiently utilize that its operation is still further removed from the maximum-efficiency point and the efficiency suffers a very marked decrease.

It will be noted also that in some cases a lamp adjusted to a rich gas shows an increased efficiency when operated on a gas of medium quality and the efficiency then decreases when a still leaner gas is used.

The curves show occasional exceptions to the general behavior in cases where a decrease in efficiency is found when using lean gas in lamps adjusted for rich gas. The explanation seems to be that the lamp was not actually adjusted to the maximum candlepower in the first place. Considerable difficulty was experienced with some lamps in getting the true maximum candlepower because of small mechanical defects in the adjusting device, which caused a shifting of the position of the gas flame within the mantle, thereby causing an abnormal variation in the candlepower in the direction of the photometer. Had it been practicable to have determined the mean-spherical candlepower directly in all cases, many of these variations might have been eliminated. The Junior burner, in particular, gave erratic results because the needle valve of the gas adjustment was not accurately centered. It was found that an accurately machined valve improved the performance of the lamp very markedly.

The following conclusions applicable in nearly all cases covered by these experiments have been drawn from them:

1. A given fluctuation in gas quality produces less change in the efficiency of operation of a lamp with lean gas than with rich gas.

2. If a lamp is adjusted for maximum candlepower, an increase in the heating value of the gas supplied to it produces a decrease in efficiency; and a decrease in the heating value produces an increase in efficiency.

7. PECULIARITIES OF INDIVIDUAL UNITS

While studying the effect of different variables on a given lamp, tests were made on different lamps using the same gas with each, and keeping all other conditions constant, to determine the comparative operation of various lamps of the same type. It was found that in many cases the difference between the individual lamps was quite as great as the effect of a changing variable. However, it was found that, in general, all lamps of a type were affected the same way by a variable and that the

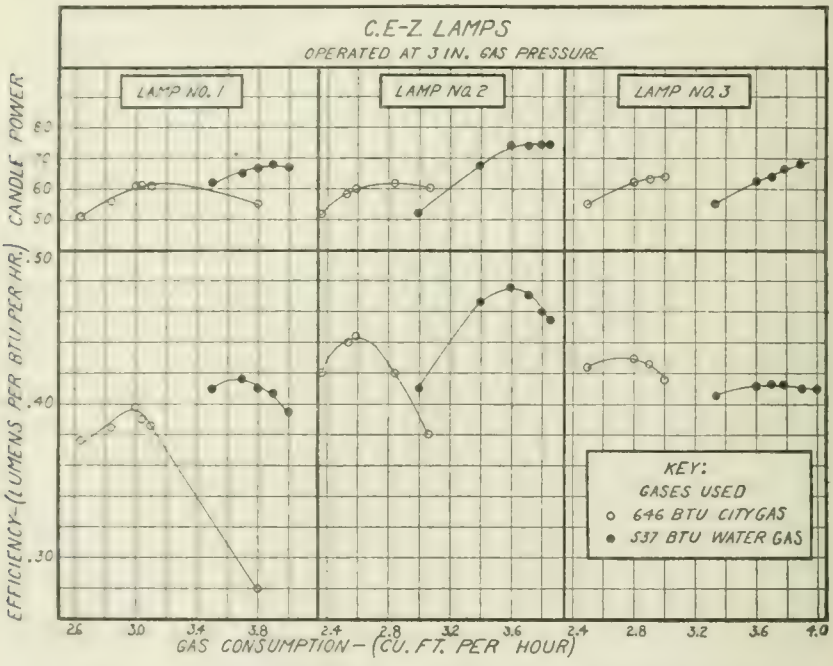


FIG. 13.—*Comparison of three similar lamps operated with the same gases*

relative effects were comparable even though the absolute values were different. The comparative operation of three C. E-Z lamps under the same conditions is shown in the curves of Fig. 13.

From these curves it will be noted that whereas two of the lamps show an increase in efficiency with the lower-quality gas, the third lamp shows slightly higher efficiency with the richer gas. It will be observed from the curves that each lamp reached a maximum efficiency and there was apparently no obstruction in any one of them preventing an adequate supply of gas. It seems quite probable that the variations are, in fact, due to some

slight differences in the sizes and shapes of the mantles on the various lamps. It has been found that there are similar differences between individual lamps of other types.

When lamps of different makers are compared there seem to be more marked differences. For example, it was found that some very cheap upright lamps gave higher efficiencies than more expensive ones, although the latter seemed to be capable of giving higher candlepowers. The causes of this behavior were not evident, though it seems likely that one cause was the different method of gas adjustment. It should be remarked that the flimsy construction of the cheap lamps might result in greatly decreased efficiency after operation for a short time.

V. GENERAL CONCLUSIONS

With uniform gas quality and with good conditions of service in other particulars the usefulness for mantle lighting of lean water gas of the qualities studied compared with rich water gas is slightly higher, for some types of lamps, than in proportion to the total heating values. Comparing lean coal gas and lean water gas of the qualities tested, the coal gas is slightly less useful, and this difference is about the same as the difference between the two qualities of water gas. Hence, it may be concluded that the usefulness of a uniform quality of coal gas of 550 to 575 Btu per cubic foot as compared with a uniform supply of richer water gas (e. g., one meeting a 22-candlepower requirement) is substantially in proportion to the heating values of the two gases. However, greater fluctuations in Btu per cubic foot, slightly greater difficulties in adjustment, and the other factors met in commercial use of lamps make the higher quality gas somewhat less desirable per heat unit for general use than either of the two kinds of lean gas.

The influence of gas quality and other factors when the gas is burned in cooking, oven-heating, and water-heating appliances will be discussed in another publication of this Bureau.

Further investigations are planned by the Bureau to extend the scope of this work and to include more unusual supplies such as blue water gas, producer gas, etc., and to obtain further general information on the subjects thus far only partly investigated.

WASHINGTON, August 5, 1917.

APPENDIX

DATA CONCERNING VARIOUS GASES USED

1. CITY GAS

Gas No.	Btu per cubic foot	Open-flame candle-power	Specific gravity	Dew point		Analysis						
				Water	Oil	CO_2	O_2	Ill.	CO	CH_4	H_2	N_2
1	648	16.8				5.4	0.2	8.9	18.4	25.8	32.5	8.8
3	664	18.6	0.63	69	48	4.5	.0	11.4	20.2	25.1	34.9	3.9
4	661	19.6	.65	65	48	2.8	.7	11.6	20.6	25.4	34.8	4.1
5	664	23.4	.66	50	47	4.4	1.3	10.9	19.4	23.7	34.3	6.0
6	662	17.2		68	50	3.7	.4	11.4	19.3	25.7	34.8	4.7
8	670	18.0	.60	68	52	2.3	.2	12.2	20.5	16.3	42.4	6.1
10	670	17.7	.60	68	52	4.0	.6	11.9	20.1	24.9	33.9	4.6
12	663	17.8	.59	66	52	4.1	.0	9.7	19.0	27.2	36.7	3.3
14	670											
16	662	18.8	.60	73	50	3.9	1.3	10.8	19.8	24.9	35.5	3.8
18	651	17.2	.60	72	48	4.2	.9	10.8	21.8	28.4	35.2	3.7
20	644	16.0	.60	71	49	4.0	1.1	10.1	20.3	24.1	36.4	4.0
22	638	15.3	.62	66	49	4.4	.8	10.0	21.9	23.2	34.5	5.2
24	640	16.3	.61	67	46	4.5	.3	10.1	21.6	24.2	34.5	4.7
26	652	16.8	.61	68	47							
28	649	15.8	.60	68	52	4.2	.5	9.7	21.0	22.5	38.1	4.0
30	665			72	53							
37	634											
43	660	18.6										
46	665											
47	626											
49	635											
52	670	18.5										
54	669	17.5										
56	670	19.0										
58	660											
60	648					4.4	.4	10.0	17.5	25.8	33.4	8.5
63	648											
64	618											
67	639											
68	640											
70	640											
71	636											
72	631											
74	631											
75	624											
75a	636											
77a	636											
88	665											
89	656											
90	654											
91	663											
94	646											
96	640											
98	646											
99	645											
101	658											
103	639											
104	686											
106	651											
108	650											
122	632	14.9										
123	633											

DATA CONCERNING VARIOUS GASES USED—Continued

2. BENZOLIZED CITY GAS

Gas No.	Btu per cubic foot	Open-flame candle-power	Specific gravity	Dew point		Analysis						
				Water	Oil	CO_2	O_2	Ill.	CO	CH_4	H_2	N_2
2	607	25.8	.....	.....	.....	1.2	1.3	13.7	16.6	23.1	39.8	4.3
7	781	25.0	.....	69	56	4.2	.5	16.4	19.3	24.6	33.6	1.4
9	728	24.0	0.64	67	53	4.3	.3	12.7	19.7	31.2	26.3	5.5
11	724	24.0	.65	61	52	4.0	.0	11.4	21.0	.....	.....	.....
13	737	25.0	.63	65	54	4.1	.3	11.4	19.9	25.3	34.2	4.8
44	695	29.1	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....
45	664	24.9	.....	.....	.....	4.4	1.1	10.8	14.5	21.1	42.8	5.3
51a	771	25.9	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....
53	753	25.8	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....
55	755	26.2	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....
121	651	20.7	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....

3. WATER GAS

15	548	10.0	0.57	65	36	4.5	0.5	8.0	24.5	18.1	40.2	4.2
17	539	7.0	.58	72	47	5.3	1.0	7.6	24.0	18.7	39.1	4.2
19	537	6.9	.59	73	49	4.9	.2	7.0	24.9	18.7	41.6	2.7
21	530	5.6	.60	68	46	4.9	.4	6.6	28.6	16.6	38.8	4.1
23	542	6.4	.63	67	48	5.9	.8	7.3	28.9	18.6	34.2	4.3
25	541	6.1	.62	65	47	5.5	.5	6.8	28.6	17.4	36.6	4.5
27	525	4.8	.....	62	46	5.7	.9	6.9	28.7	16.7	37.6	3.5
29	551	6.8	.61	.....	.....	5.0	.4	7.5	26.9	13.6	46.4	4.0
41	523	.....	.....	.....	.....	4.5	1.0	9.2	21.3	17.4	41.4	5.2
42	487	.....	.....	.....	.....	6.1	.9	6.2	24.1	.....	.....	.....
57	575	13.4	.....	.....	.....	3.2	.1	4.6	27.3	18.5	40.6	5.6
59	565	.....	.....	.....	.....	5.1	.1	7.1	24.5	19.7	37.6	5.9
61	561	.....	.....	.....	.....	3.0	1.1	6.8	23.6	19.3	41.3	4.9
65	580	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....
66	553	.....	.....	.....	.....	3.0	1.4	6.8	28.6	18.1	36.0	6.1
69	550	.....	.....	.....	.....	3.2	2.6	6.4	25.7	17.7	29.9	14.5
73	550	.....	.....	.....	.....	4.1	1.5	7.5	19.9	21.1	40.1	5.8
76	554	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....
77	548	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....
79	567	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....
80	590	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....
81	544	.....	.....	.....	.....	3.5	.8	7.4	28.3	20.5	33.7	5.8
83	567	.....	.....	.....	.....	3.3	.8	8.1	30.2	33.8	19.1	4.6
86	570	.....	.....	.....	.....	4.1	4.2	5.9	24.3	17.1	44.0	.4
87	566	.....	.....	.....	.....	3.4	.6	7.4	31.8	20.8	32.7	3.3
92	580	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....
93	574	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....
95	561	.....	.....	.....	.....	3.2	2.1	7.0	32.2	16.8	29.7	9.0
97	537	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....
100	567	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....
102	573	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....
105	570	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....
107	554	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....
109	561	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....
110	558	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....
116	582	.....	.....	.....	.....	1.8	.6	7.5	25.9	18.0	37.7	8.5
118	575	.....	.....	.....	.....	2.4	1.0	7.1	27.3	19.0	35.6	7.6

4. COAL GAS

38	575	.....	.....	.....	.....	4.0	0.9	4.1	5.1	28.6	53.9	3.4
39	592	.....	.....	.....	.....	3.8	1.1	3.2	5.6	31.8	52.2	2.3
40	572	.....	.....	.....	.....	4.9	.3	3.0	4.6	31.8	52.6	2.8
62	561	.....	.....	.....	.....	6.3	.4	6.1	8.2	25.5	47.0	6.5
78	540	.....	.....	.....	.....	3.2	3.1	3.1	7.3	28.9	39.0	15.4
82	570	.....	.....	.....	.....	4.2	.7	3.6	9.3	33.0	46.7	2.5
84	540	.....	.....	.....	.....	9.0	3.4	4.7	7.1	27.7	41.4	6.7
85	564	.....	.....	.....	.....	5.5	.8	3.1	8.5	32.3	44.4	5.4
111	573	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....
117	576	.....	.....	.....	.....	2.7	.0	3.7	7.0	22.1	57.8	6.7
119	575	.....	.....	.....	.....	3.4	.5	4.5	11.3	19.1	53.2	8.0
120	580	.....	.....	.....	.....	2.2	1.0	3.0	7.5	30.0	48.7	7.6

DEPARTMENT OF COMMERCE

TECHNOLOGIC PAPERS

OF THE

BUREAU OF STANDARDS

S. W. STRATTON, DIRECTOR

No. 111

COMPRESSIVE STRENGTH OF LARGE BRICK PIERS

BY

J. G. BRAGG, Assistant Physicist

Bureau of Standards

ISSUED SEPTEMBER 20, 1918

PRICE, 10 CENTS

Sold only by the Superintendent of Documents, Government Printing Office
Washington, D. C.

WASHINGTON
GOVERNMENT PRINTING OFFICE
1918

COMPRESSIVE STRENGTH OF LARGE BRICK PIERS

By J. G. Bragg

CONTENTS

	Page
I. Introduction	4
II. Review of previous tests	5
1. Howard's tests	5
2. McCaustland's tests	6
3. Macgregor's tests	7
4. Kreuger's tests	8
III. Scope of investigation	10
1. Purpose	10
2. Outline of program	10
(*A*) Original program	11
(*a*) Districts	11
(*b*) Bricks	11
(*c*) Mortars	11
(*d*) Bond and workmanship	12
(*B*) Revised program	11
(*a*) Districts	12
(*b*) Bricks	12
(*c*) Mortars	13
(*d*) Bond and workmanship	13
3. Supplementary tests	14
(*A*) Piers	14
(*B*) Individual bricks	14
IV. Laboratory procedure	14
1. Construction of piers	14
2. Testing	14
(*A*) Individual bricks	14
(*a*) Transverse tests	14
(*b*) Compression tests	15
(*c*) Absorption tests	15
(*B*) Piers	15
(*a*) Machine used	15
(*b*) Placing piers in machine and capping	16
(*c*) Observations	18
(*d*) Instruments used	18
V. Results of tests	19
1. Observed data	19
2. Derived data	26
(*A*) Relation of transverse strength of bricks to compressive strength of piers	26
(*B*) Relation of compressive strength of bricks to compressive strength of piers	28
(*C*) Modulus of elasticity	30
(*D*) Lateral strains	31

Page

VI. General discussion 32
1. Type of failure 32
2. Effect of quality of bricks 35
3. Effect of mortars 36
4. Effect of bond 36
VII. Summary 38

I. INTRODUCTION

The compressive strength of brick piers has been the subject of several important investigations. Until quite recently, however, testing machines of sufficient capacity had not been available for use in studying the deportment of test specimens comparable in size with those entering into the erection of large structures. A majority of the investigations of the load-bearing value of brick masonry have therefore been conducted on comparatively small specimens. In general, considerable difficulty is experienced in applying laws deduced from model tests in the laboratory to the problems confronting the engineer in practice. This is especially true of masonry, in which the conditions governing erection vary to a great extent in different districts. The few tests which have been made on large piers were upon specimens of which the conditions pertaining to their erection were ideal, even to the extent that in some cases slightly warped bricks were discarded. Other refinements were introduced, such as special screening of the sand used in the mortar in order to obtain the thinnest possible joints. Such tests are very necessary in the study of certain definite laws. However, due allowance must be made for the conditions under which the tests were conducted, since these conditions are not realized in practice.

In 1914 the technical committee of the National Brick Manufacturers' Association, recognizing the need for a more comprehensive investigation of the strength of brick masonry, outlined a series of tests of large brick piers. These tests have been conducted under a cooperative arrangement between the Bureau of Standards and the National Brick Manufacturers' Association and form the basis of this report. It is believed that the results obtained are of general interest, as the piers tested were of a size commensurate with engineering practice, and the mortars and grade of workmanship applied to their construction were such as would be expected in ordinary building practice. The investigation is composed of tests on piers 2 feet 6 inches square by 10

feet high, in which three grades of brick were used. These bricks were representative of four widely separated districts east of the the Mississippi River. Three mortars were used in the beginning and three bonds, representative of three grades of workmanship, were used throughout the investigation. Transverse, crushing, and absorption tests were made on the individual bricks and a few supplementary pier tests were made, in which wire meshing was used to determine the influence upon the bond.

II. REVIEW OF PREVIOUS TESTS

The following investigations have been carefully considered in the preparation of this report and will be referred to from time to time throughout the discussion.

1. HOWARD'S TESTS[1]

This work consists of several series of pier tests, in which a study was made of various mortars, grades of brick, and methods of laying the bricks. A novel feature of this work was the laying of bricks on edge and in some cases breaking joints every third or sixth course, instead of every course. The piers tested ranged in cross sectional dimensions from 8 inches by 8 inches to 16 inches by 16 inches, the heights varying from 2 feet to 12 feet 6 inches. In these tests, 14 of which were made on face-brick piers laid in 1 part Rosendale cement to 2 parts sand mortar, the strength was found to vary with the height of pier, the ultimate resistance of the pier varying from 12.5 to 18.1 per cent of the compressive strength of the bricks. Thirty-eight common-brick piers of the same mortar and general dimensions developed a strength of from 7.8 to 17.6 per cent of the compressive strength of the bricks. Laying the bricks on edge and breaking joints every third or sixth course increased the strength considerably.

Results of some of these tests are given in the following table, which is taken from Burr's Elasticity and Resistance of the Materials of Engineering, sixth edition, page 426.

[1] "United States report of tests of metals and other materials, 1884-1891," Engineering Record, March 22, 1913; Clay Worker, March 1913

TABLE 1.—Crushing Strength of Brick Piers of Various Sizes laid in Different Mortars

No.	Height of pier	Section of pier	Composition of mortar	Weight per cubic foot	Ultimate resistance
	Ft. in.	Inches		Pounds	Lbs. /in.[2]
[a] 1	1 4	8 by 8	1 lime, 3 sand	137.4	2520
[a] 2	6 8	8 by 8	do	133.5	1877
[a] 3	1 4	8 by 8	1 Portland cement, 3 sand	136.3	3776
[a] 4	6 8	8 by 8	do	133.5	2249
[a] 5	2 0	12 by 12	1 lime, 3 sand		1940
[a] 6	2 0	12 by 12	do		1900
[a] 7	10 0	12 by 12	do	131.7	1511
[a] 8	10 0	12 by 12	do	125.0	1807
[a] 9	2 0	12 by 12	1 Portland cement, 2 sand		3670
[a] 10	10 0	12 by 12	do	132.2	2253
[b] 11	1 4	8 by 8	1 lime, 3 sand	135.6	2440
[b] 12	6 8	8 by 8	do	133.6	1540
[b] 13	2 0	12 by 12	do		2150
[b] 14	2 0	12 by 12	do		2050
[b] 15	9 9	12 by 12	do	131.5	1118
[b] 16	10 0	12 by 12	do	136.0	1587
[b] 17	10 0	12 by 12	1 Portland cement, 2 sand	131.0	2003
[b] 18	2 8	16 by 16	do		2720
[b] 19	10 0	16 by 16	do		1887

[a] The kind of brick used in this test was face brick, with an average compressive strength of 13 925 pounds per square inch.

[b] The kind of brick used in this test was common brick, with an average compressive strength of 18 337 pounds per square inch.

2. McCAUSTLAND'S TESTS[2]

This investigation was composed of a series of 14 piers 13 inches by 13 inches in cross sectional dimensions and 80 inches high, which were reinforced laterally in the horizontal joints with steel plates, straps, or wire meshing. The mortar was composed of 1 part Portland cement and 3 parts sand. The bricks used had a compressive strength of 3500 pounds per square inch. In these tests it is shown that the efficiencies of the piers reinforced with iron straps and plates are less than those of the piers without reinforcement. The piers reinforced with wire mesh in every joint developed efficiencies of 46 per cent as compared with 30 per cent for those without reinforcement. However, there is a considerable drop in efficiency from the piers with wire mesh in every joint and piers with wire mesh in every second joint, which developed efficiencies of only 33 per cent.

[2] E. J. McCaustland, Transactions of the Association of Civil Engineering of Cornell University for 1900.

The following table of results of McCaustland's tests is taken from Burr's Elasticity and Resistance of the Materials of Engineering, sixth edition, page 425.

TABLE 2.—Ultimate Compressive Resistance of 13 by 13 by 80 Inch Brick Piers with Metal Reinforcement in Horizontal Joints

[Built with 1:2 Portland cement mortar; joints 0.3 inch thick]

No.	Kind of joints	Ultimate stress		Efficiency per cent of single brick	No.	Kind of joints	Ultimate stress		Efficiency per cent of single brick
		Total	Pounds per square inch				Total	Pounds per square inch	
1	Portland-cement mortar 1:2	194 000	1150	30	7	Wire netting every second course	192 000	1136	33
2	do	200 000	1184		8	do	208 000	1248	
3	Iron straps every fourth course	136 400	810	24	9	Wire netting every course	282 000	1694	46
4	do	155 400	920		10	do	240 000	1440	
5	Iron straps every sixth course	130 000	780	22	11	Iron plate every fourth course	174 000	1030	
6	Iron straps every eighth course	142 500	843	24	12	do	193 500	1145	28
					13	do	162 000	974	
					14	do	143 000	858	

3. MACGREGOR'S TESTS[3]

The object of this investigation was to ascertain what proportions of cement and lime may be used to advantage in a cement-lime mortar and the effect of such mortars on the ultimate compressive strength of brick masonry. Seven sets of brick piers 8 inches by 8 inches by 84 inches were tested. Each set of nine piers represented a different proportion of mortar with varying amounts of hydrated lime. Each set was composed of three groups of three piers each, group 1 being tested at 7 days, group 2 at 28 days, and group 3 at 90 days. The bricks used were hard-burned face bricks. There were also tested seven piers of common brick, one for each different mortar. These piers served as a check on the hard-burned face brick piers, and were tested at the age of 28 days. In these tests piers laid in a mortar composed of 1 part (25 per cent lime and 75 per cent Portland cement) to 3 parts sand by volume developed the highest strength. Piers laid in mortar composed of 1 part (50 per cent lime and 50 per cent Portland cement) to 3 parts sand developed higher strengths than piers laid in 1 part cement to 3 parts sand mortar.

[3] Tests made by Prof. J. S. Macgregor, Columbia University; Bulletin J, Hydrated Lime Bureau of the National Lime Manufacturing Association.

The following data are taken from Bulletin J, Hydrated Lime Bureau of the National Lime Manufacturing Association:

TABLE 3.—Effect of Cement-Lime Mortar on Strength of 8 by 8 Inch Brick Piers

Mortar mixture used		Age when tested	Compressive strength, pounds per square inch	
By volume	By weight		Face-brick piers; each result an average of 3 tests	Common-brick piers; 1 test only
		Days		
1 Portland cement; 3 sand	100 Portland cement; 300 sand	7	2630	
		28	2840	1170
		90	2840	
0.90 Portland cement; 0.10 hydrated lime; 3 sand.	90 Portland cement; 4 hydrated lime; 300 sand.	7	3080	
		28	3170	1189
		90	4435	
0.85 Portland cement; 0.15 hydrated lime; 3 sand.	85 Portland cement; 6 hydrated lime; 300 sand.	7	2890	
		28	3230	1340
		90	4300	
0.75 Portland cement; 0.25 hydrated lime; 3 sand.	75 Portland cement; 10 hydrated lime; 300 sand.	7	3120	
		28	3470	1685
		90	4170	
0.50 Portland cement; 0.50 hydrated lime; 3 sand.	50 Portland cement; 20 hydrated lime; 300 sand.	7	2760	
		28	3100	1300
		90	3820	
0.25 Portland cement; 0.75 hydrated lime; 3 sand.	25 Portland cement; 30 hydrated lime; 300 sand.	7	1945	
		28	2370	1032
		90	2720	
1 hydrated lime; 3 sand	40 hydrated lime; 300 sand	7	1535	
		28	1870	
		90	1950	

4. KREUGER'S TESTS[4]

A recent investigation by Prof. H. Kreuger at the Technical High School in Stockholm, although conducted on small piers, is quite comprehensive in its scope. This investigation includes most of the variables referred to in previous tests and some tests were made to study the effect of eccentric loading. The piers tested were approximately 11 inches square, ranging in height from 6 inches to 33 inches. With bricks of various strengths laid in 1 part lime to 3 parts sand mortar the piers developed strengths of from 18.5 to 26.5 per cent of the ultimate compressive strength of the bricks. It must be noted, however, that the results obtained by Prof. Kreuger are not comparable with results of tests made in the United States, since the method of testing the individual bricks is different. Prof. Kreuger's results were obtained from compression tests on halves of the same brick cemented

[4] Tonind-Ztg 40, 1916; Clay Worker, July, 1916, and August, 1916.

together. Since the compressive strength developed in this manner would be considerably lower than in the case of a single half brick tested flat, the efficiency of the pier would be correspondingly higher. Tests of the mortars used showed an increase in strength from 28 days to 1 year of 33 to 165 per cent, while the piers increased in strength in the same length of time only 6 to 17 per cent. The introduction of wire mesh in every joint increased the strength 88 to 100 per cent. Piers loaded eccentrically on one-half their bearing surfaces failed at loads slightly under one-half the loads sustained by piers loaded concentrically, and no cracking was observed on the so-called tension side of the pier.

The following data are taken from the Clay Worker for July, 1917, and August, 1917. The results have been converted from the metric to the English units of measure and retabulated.

TABLE 4.—Influence of the Strength of Brick

[Mortar: 1 lime, 3 sand. Age, 28 days]

Number of piers tested	Height of piers	Breadth of piers	Average compressive strength of—		Per cent compressive strength of bricks developed in piers		
			Brick used	Piers	Minimum	Maximum	Average
	Inches	Inches	Lbs./in.²	Lbs./in.²			
5	34.0	10.0	1920	410	19.3	23.7	21.3
4	34.0	9.5	2510	670	23.7	29.5	26.5
3	33.0	9.5	4040	880	20.0	24.3	21.8
5	33.5	9.5	5300	980	15.0	21.2	18.5
4	31.0	9.5	7120	1820	24.7	26.2	25.5
5	30.5	9.0	8600	1680	17.5	21.6	19.5

TABLE 5.—Influence of the Strength of Mortar

[Mortar: 1 lime, 3 sand. Age, 28 days]

Pier No.	Compressive strength of		Mortar mixture	Compressive strength of piers
	Bricks	Mortar		
	Lbs./in.²	Lbs./in.²		Lbs./in.²
1	4040	0	Dry sand	740
2	4040	38	1 lime; 3 sand	740
3	4040	355	2 lime; 1 cement; 9 sand	1420
4	4040	695	1 lime; 1 cement; 6 sand	1840
5	4040	1280	1 lime; 2 cement; 9 sand	1700
6	4040	1640	2 lime; 1 cement; 7 sand	1930
7	4040	2620	1 cement; 3 sand	1980

TABLE 6.—Influence of Varying the Height of the Piers

[Mortar 1 lime, 3 sand]

Pier No.	Compressive strength of bricks	Breadth of pier	Ratio, height to breadth	Compressive strength of piers	Pier No.	Compressive strength of bricks	Breadth of pier	Ratio, height to breadth	Compressive strength of piers
	Lbs. in.²	Inches		Lbs. in.²		Lbs./in.²	Inches		Lbs./in.²
1	3260	10.6	4.3	2340	10	3260	10.6	43.4	880
2	3260	10.6	8.7	2320	11	3260	10.6	48.8	750
3	3260	10.6	13.0	1940	12	3260	10.6	53.2	780
4	3260	10.6	17.4	1620	13	3260	10.6	57.5	640
5	3260	10.6	21.7	1090	14	3260	10.6	61.8	610
6	3260	10.6	26.4	1020	15	3260	10.6	65.8	660
7	3260	10.6	30.7	Broken.	16	3260	10.6	69.7	660
8	3260	10.6	35.4	780	17	3260	10.6	74.5	610
9	3260	10.6	39.5	880	18	3260	10.6	78.8	610

TABLE 7.—Effect of Eccentric Loading

[Mortar 1 lime, 3 sand]

Height of pier	Breadth of pier	Compressive strength of brick	Compressive strength of mortar	Maximum load	
				Concentric load	Eccentric load
Inches	Inches	Lbs./in.²	Lbs./in.²	Lbs./in.²	Lbs./in.²
33	9.4	4040	50	890	417
33	9.4	5600	50	1220	570
31	9.4	7100	50	1850	850

III. SCOPE OF THE INVESTIGATION

1. PURPOSE

The purpose of this investigation was to determine the strength developed by brick piers of normal size as used in modern buildings, using in their construction such materials and grades of workmanship as are available in the United States.

The data contained in previous studies along this line were in a measure incomplete or represented conditions more favorable than may be realized in practice.

2. OUTLINE OF PROGRAM

The chief variables considered in the original outline of the investigation were (1) the quality of bricks employed with respect to grade and geographical location; (2) the quality and kind of mortar; (3) the grade of workmanship employed; and (4) the bonding of courses or method of laying the bricks.

These were considered the most important variables among the many which enter into an investigation of this nature. The

consideration of minor variables on piers of the size included in the present investigation is not only prohibitive as to cost, but for the most part they may be studied to better advantage on smaller specimens.

The original program included tests of 144 piers. As the work progressed this program was modified to a great extent, as it was found that certain of the variables under consideration did not yield data of sufficient importance to justify the expenditure necessary in the erection and testing of large piers.

(*A*) ORIGINAL PROGRAM.—(*a*) *Districts.*—The bricks were to be selected from four districts east of the Mississippi River, as follows:

Eastern district, A: New York and adjacent territory in New Jersey or eastern Pennsylvania.

Eastern central district, B: Western Pennsylvania and eastern Ohio, comprising Pittsburgh and points within a 50-mile radius.

Western central district, C: Chicago and Galesburg, Ill.

Southern district, D: Atlanta, Birmingham, and New Orleans.

(*b*) *Bricks.*—From each district there were to be selected four grades of brick corresponding with those of Committee C–3, American Society of Testing Materials, tentative specifications 1913. These grades are defined in the following statement:

Class A: Vitrified. Average unit compressive strength at least 5000 pounds per square inch. Average absorption not more than 5 per cent.

Class B: Hard burned. Average unit compressive strength at least 3500 pounds per square inch. Average absorption not more than 12 per cent.

Class C: Common firsts. Average unit compressive strength at least 2000 pounds per square inch. Average absorption not more than 18 per cent.

Class D: Falling below the above specifications.

(*c*) *Mortars.*—Three piers each of three different mortars were to be tested for each grade of bricks used. The following mortars were specified:

A. A pure Portland cement and sand mortar to be composed of 1 part cement to 3 parts sand.

B. A dolomitic lime mortar tempered with Portland cement to be composed of 1 part lime hydrate to 3 parts sand tempered with 5 per cent (dry weight) Portland cement.

C. A lime mortar free of Portland cement to be composed of 1 part lime to 3 parts sand.

(d) *Bond and Workmanship.*—Each group of three piers was to be representative of the following three grades of bonding and workmanship:

Grade A: Every other course to be a header course and all joints squeeze joints except inclosures where the joints must be thoroughly filled after the bricks are in place. Each course to be finished complete before beginning the next.

Grade B: Every fourth course to be a header course. Center joints to be filled as thoroughly as practicable without squeezing. Each course to be finished up complete before beginning the next.

Grade C: Every seventh course to be a header course and no squeeze joints used. Outer rim of pier to be built up three or four courses and the center filled in, one course at a time, with loose bricks and slushed with mortar for the bedding of the next course. No individual points to be filled except as they will fill by slushing.

(*B*) Revised Program.—The original program specified a total of 36 piers from each of the four districts, but revisions were made on the basis of results obtained in the early stages of the investigation, which reduced the total number of piers tested from 144 to 50. As the work was begun on materials from the Pittsburgh district and the revisions in the program were based on these tests, the piers of this district more nearly conform to the original program than do those of the other three districts. As a result of the revisions made in the original program, the piers of the Pittsburgh district comprise something over 50 per cent of the entire investigation.

(a) *Districts.*—No important changes were made in the matter of districts. It was found to be impracticable, however, to cover the entire field in each district, and with the exception of the Pittsburgh district the bricks obtained from each locality represent the product of a single manufacturer. It is believed, however, that although the geographical locations do not conform in detail with the original program, they are in close enough agreement to be fairly representative of the product east of the Mississippi River.

(b) *Bricks.*—The low strengths developed in the piers built of C-grade bricks, as defined in the original program, did not seem to warrant the use of a fourth grade. It was therefore decided to confine the tests to the first three grades only. From the tests of individual bricks selected at random from the materials received at the laboratory, it was found that none of the grades more than

approximated the requirements as outlined in the tentative specification of the American Society for Testing Materials. In one instance where it was necessary to order a second lot of bricks from a manufacturer in district B, the piers of the second lot developed a strength of only one-half those of the first lot. It was decided, therefore, to grade the bricks from each district as follows:

Grade 1: Hard burned or best quality.

Grade 2: Medium burned or considered as common.

Grade 3: Soft burned or poorest product marketed.

In the Chicago district only two grades of brick were obtained, with but a slight difference in the two grades. Only enough material for two piers instead of three was obtained for two of the grades in the New Orleans district. Since the piers from this district were last to be tested it was thought best, after consideration of the trouble experienced in obtaining materials, to finish

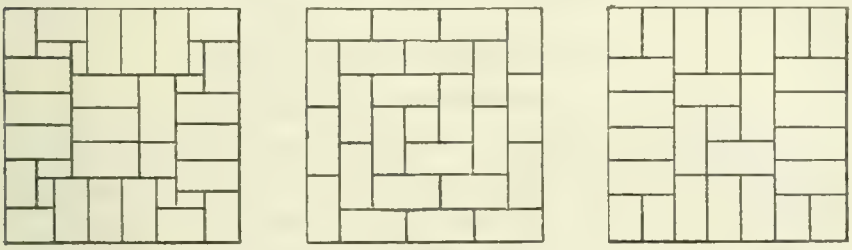


FIG. 1.—*Arrangement of bricks in courses*

the investigation without the two piers needed to complete this district.

(*c*) *Mortars.*—The following mortars were used after consideration of the results obtained on the first few piers of the Pittsburgh district:

Cement mortar: One part pure Portland cement to 3 parts sand.

Lime mortar: One part hydrated lime to 6 parts sand.

Cement-lime mortar: One part composed of 15 per cent hydrated lime (dry weight) and 85 per cent Portland cement (dry weight) to 3 parts sand by weight.

(*d*) *Bond and Workmanship.*—In the case of bond and workmanship no changes were introduced in the method of bonding, the general procedure as outlined in the original program being carried on throughout the investigation. It is believed, however, that the grade of wormanship is somewhat superior to what might be expected in practice. The arrangement of bricks in the header and stretcher courses is shown in Fig. 1.

3. SUPPLEMENTARY TESTS

While no supplementary tests were specified in the original program, numerous tests have been made in order to more closely define the strengths due to the quality of bricks used and the method of laying them in the piers.

(*A*) PIERS. The results of the earlier tests indicated a falling off in strength as the number of headers were increased. As this is contrary to the accepted theory that the effect of "tying in" the masonry with header courses is to increase the strength, some tests were made to further determine the action of bond. Four piers of the same cross-sectional dimensions as those included in the main investigation and 5 feet in height were built, introducing a different arrangement of the bricks in the header course and the use of wire mesh in the horizontal joints.

(*B*) INDIVIDUAL BRICKS.—Tests of individual bricks were made according to the methods commonly employed in testing laboratories.

IV. LABORATORY PROCEDURE

1. CONSTRUCTION OF PIERS

The piers were built and stored in the laboratory until tested. For convenience in handling they were built on steel base plates 36 inches square by 1 inch thick. As these plates were not removed during the test they were plane surfaced on the side in contact with the testing-machine platen. The plates were carefully leveled on the floor before starting the piers, and the piers were plumbed during erection so that the vertical axis of the finished piers was perpendicular to the plane surface of the steel base plate. The piers were built by contract, and the time necessary to build a single pier varied from one to two days. They were uniformly 10 feet high and the number of courses varied from 41 to 46, depending upon the thickness of the bricks. The horizontal joints varied in thickness from three-eighths to one-half inch.

2. TESTING

(*A*) INDIVIDUAL BRICKS.—No special refinements were introduced in the tests of individual bricks, for it was thought better in view of the practical nature of the investigation to make a more than usual number of tests on each lot of brick in order to obtain a fair average.

(*a*) *Transverse Tests.*—Twenty transverse tests were made on each lot of bricks received, except in the case of grade 3, from

the Pittsburgh district. Of this lot only enough material was available for 10 tests. No bricks were culled because of warpage or cracks due to burning. The tests were made on a small Olsen hand-power machine of 10 000 pounds capacity. Each sample was supported at the ends on round steel bars 1 inch in diameter. These bars were spaced 7 inches, center to center, making a 7-inch span for all tests. Because of the warped condition of certain samples it was necessary in some cases to place fillers between the roller and the brick at one end of the roller. The load was applied at the center of the 7-inch span through a small adjustable hemispherical bearing block resting on a three-fourths-inch diameter round steel rod. From these tests the moduli of rupture were computed by the formula:

$$R = \frac{3\,wl}{2\,bd^2}, \text{ in which}$$

R = modulus of rupture,
w = breaking load,
l = span in inches,
b = breadth in inches,
d = depth in inches.

(*b*) *Compression Tests.*—Of the half bricks left from the transverse tests 10 samples were tested on edge and 10 samples were tested flat. Specimens were selected which had broken through the center in the transverse test, so that the sizes were approximately the same. All samples were capped top and bottom with plaster of Paris, with as nearly as possible uniform thickness of cap. Lack of parallelism of the two bearing surfaces was compensated for in the testing machine by an adjustable hemispherical bearing block.

(*c*) *Absorption Tests.*—Absorption tests were made on the samples to be tested in compression. The samples were first placed in the drying oven and dried to a constant weight, after which they were immersed in water for 48 hours and again weighed. The percentage absorption was then calculated in terms of the dry weight as follows:

$$\text{Per cent absorption} = \frac{w_1 - w}{w}, \text{ in which}$$

w = dry weight,
w_1 = the weight after 48 hours immersion in water.

(*B*) PIERS.—(*a*) *Machine Used.*—The piers were tested on the 10 000 000-pound Olsen hydraulic testing machine. This machine

is equipped for compression tests only and is of the vertical type, having a length capacity of 25 feet. The upper platen may be moved up or down on the four pulling screws for adjustment to the length of specimen and is actuated by a 15 horsepower electric motor mounted on top of the platen. The lower platen rests upon a hemispherical base having a 5-foot radius of curvature. The whole rests on a piston 50 inches in diameter, which is actuated by a triple-plunger pump mounted on a tank containing a supply of oil, which is forced into the cylinder under the lower platen. The tank forms the base for a standard Olsen lever system. The pressure in the cylinder is communicated to the lever system through a smaller piston having a knife-edge bearing on the main lever. By this method about one-eightieth of the total load on the machine is weighed. The scale beam, however, is graduated to read direct the total load on the machine. At the capacity of the machine, which is 10 000 000 pounds, the oil pressure in the cylinder is 5 000 pounds per square inch. The machine, exclusive of the pump and lever system, is shown in Fig. 2.

(*b*) *Placing Piers in Machine and Capping.*—The piers were not moved from the position in which they were built until they were aged sufficiently for test. This was four months after building for the lime mortar piers, and one month after building for the cement and cement-lime mortar piers. A steel plate 36 inches square by 1½ inches thick, with suitable eyebolts for crane-hook connection, was placed on top of the pier and connected to the steel base plate by four vertical steel rods. The pier was then moved to the testing machine with an electrical crane and transferred to a smaller auxiliary crane attached to the upper platen of the testing machine. The pier was then lowered gently into place after appropriate centering with the vertical axis of the machine. This was accomplished in all cases without damage to the pier. The upper plate and rods were then removed and the lower platen of the machine was raised so that the combined weight of the pier and platen rested on the oil. The upper platen was then lowered to within a few inches of the pier and the lower platen tipped on its adjustable base until the top of the pier was parallel to the upper platen. Plaster of Paris was then placed on the top of the pier and the upper platen brought down until the superfluous plaster was squeezed out. This left a cap having a uniform thickness of from one-fourth to three-eighths inch.

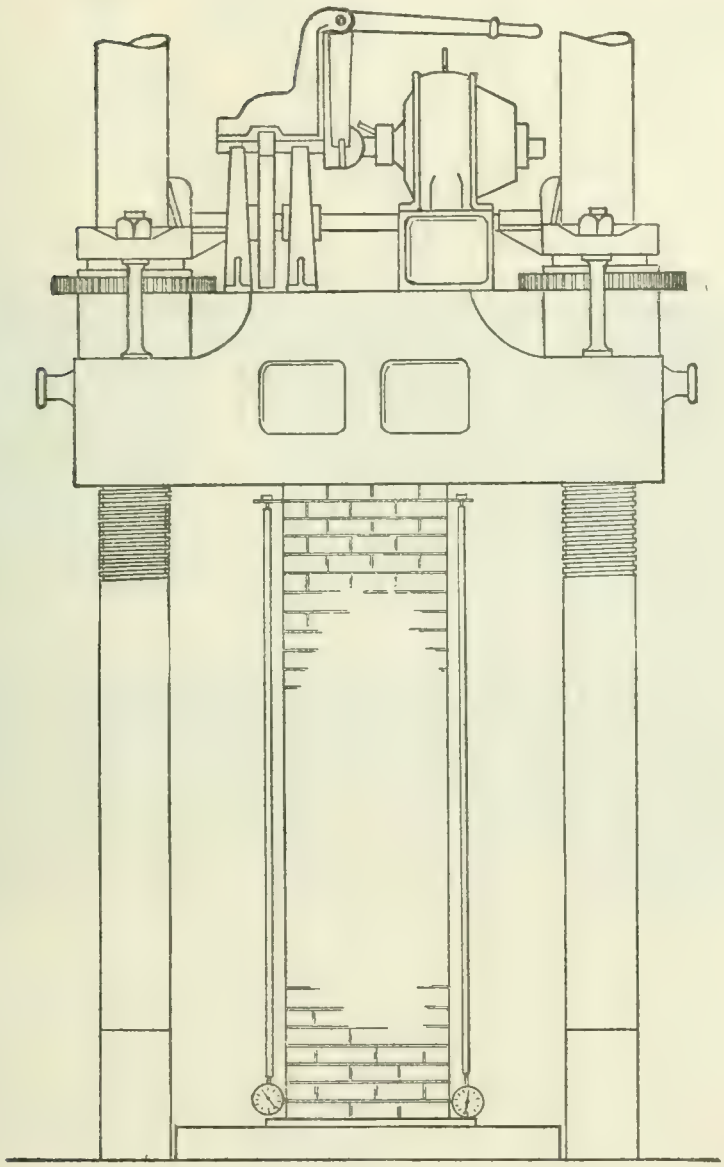


FIG. 2.—*Diagrammatic view of pier in testing machine*

(*c*) *Observations.*—Two sets of observations were made on each pier, one set measuring the total compression from top to bottom courses and the other set measuring the compression and lateral strain in short-gage lengths at the center of the pier. For the total compression or "full-length" compression readings steel plates one-fourth by 1 by 6 inches were inserted in the first and last joints at the corners of each pier at the time of building. Holes were bored in the projecting end of these plates to which were attached adjustable brass rods and Ames gages. The Berry strain gage was used for all measurements of short-gage length. For this purpose three-sixteenths inch German silver plugs were inserted in holes in the brick and set in plaster of Paris. The strain gage points were seated in No. 55 drill holes in the plugs. The holes in the bricks were drilled with a one-half-inch stone-cutter's tool, a small air hammer of high speed but very little hitting force. It was found that a slower hammer of greater hitting force had a tendency to loosen the bricks in the mortar.

(*d*) *Instruments Used.*—The apparatus used to measure full-length compression consists of four brass rods, one for each corner of the pier, and four Ames gages of the same type used on the Berry strain gage shown in Fig. 3. The rods are adjustable to length and vertical alignment and are attached by set screws to the steel plates in the top joint of the pier. They are fitted with steel points at the lower end, which are seated in holes bored in the Ames gage spindles. Calibrations of the Ames gages show a possible error of 0.001 inch. The gage length used, however, was 114 inches and in consideration of the amount of compression for each increment of load an error of 0.001 inch is small enough to be negligible.

The Berry strain gage used was calibrated by means of the apparatus shown in Fig. 3. This apparatus consists of a square steel bar, one end of which is fitted with a movable steel plug actuated by a carefully calibrated Brown & Sharpe micrometer gage head reading to 0.0001 inch. The steel plug is very carefully fitted and has a lug attached to it, which moves in a slot in the top of the steel bar. The lug serves the purpose of keeping the plug in the same relative position for each series of readings and also furnishing the surface in which are bored the drill holes for the movable leg of the strain gage. The following three sizes of holes are bored in the lug; No. 54 diameter, 0.055 inch; No. 55 diameter, 0.052 inch; No. 56 diameter, 0.046 inch. Drill

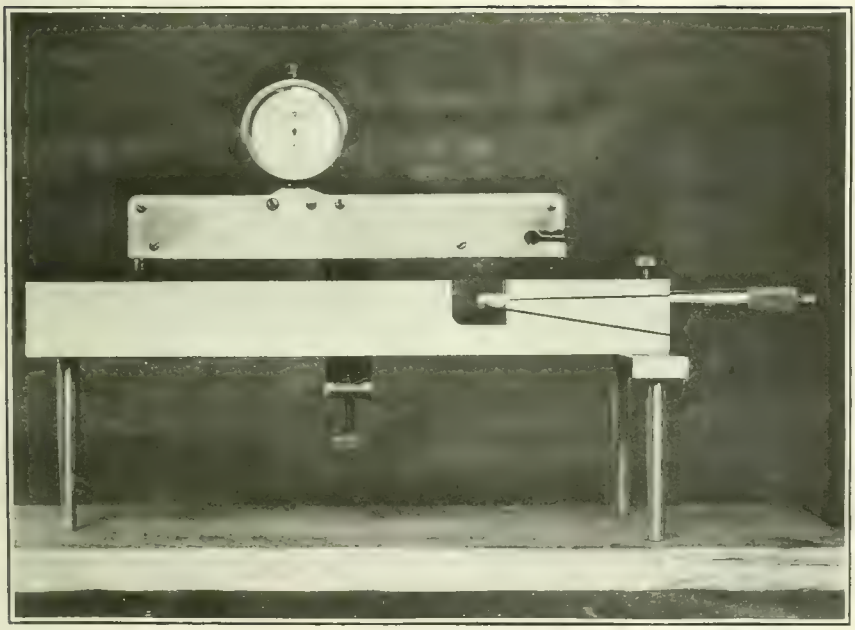


FIG. 3.—*Apparatus for calibrating Berry strain gage showing gage in position for calibration*

holes are placed in the main part of the square bar at proper distances from the lug so that Berry gages from 2 to 10 inch gage lengths may be calibrated. An error is introduced in the Berry gage readings, due to the seating of the movable leg in the drill holes. The effect is the same as would be produced by shortening the movable leg of the gage and changes the multiplying ratio so that a different correction constant must be used for each size of drill hole. To determine this constant with the apparatus shown in Fig. 3 requires very little time, and no special

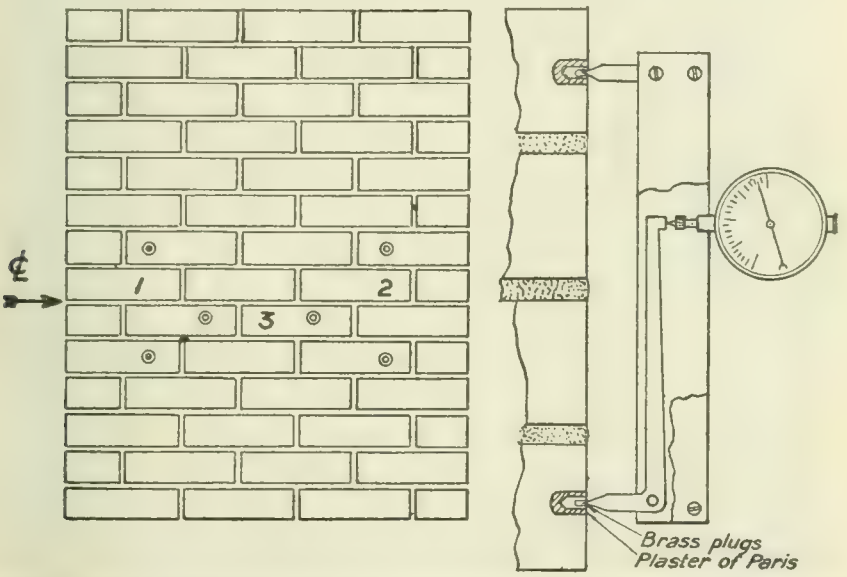


FIG. 4.—*Showing location of Berry strain gage stations on each face of pier and method of seating gage points*

care need be exercised regarding changes of temperature. A sketch showing the locations of the Berry gage observation stations, the method of inserting the plugs, and the position of the gage is shown in Fig. 4.

V. RESULTS OF TESTS

1. OBSERVED DATA

In view of the large mass of data contained in the original log sheets, the data have been grouped in tables, and only in those cases which are characteristic of types will the original logs be presented. The data obtained are summarized in Tables 8 and 9. These tables include details of construction, age, ultimate

stress, and modulus of elasticity for each pier, also the average strengths and percentage of absorption of the bricks used in their construction. The strengths of bricks and piers included in Table 8 are shown in Fig. 5. The lines connecting the points in this diagram indicate how the strength of the piers follow the strengths of the individual bricks. Table 10 is a copy of

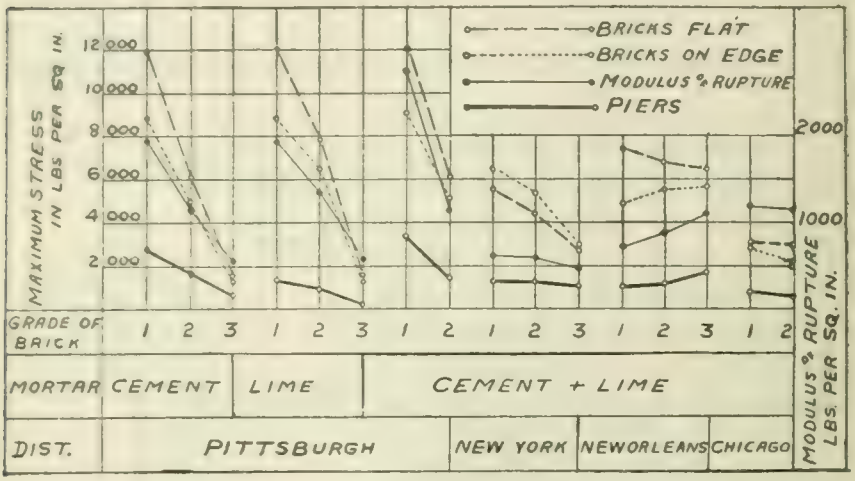


FIG. 5.—*Strength of piers and individual bricks shown diagrammatically*

See Table 1 for exact values

the original log for pier B 30, which is typical of the entire investigation. In Table 11 is shown the log of grade 1 bricks from the Pittsburgh district. Photographs of piers after failure are shown in Figs. 6 to 14. In these photographs is shown the type of failure characteristic of all piers tested, the vertical cracking of the masonry being very pronounced in all cases.

FIG. 6.—*Pier B 14, Serial No. 2. Grade 1 bricks from Pittsburgh district laid in cement mortar*

FIG. 7.—*Pier B 31, Serial No. 10. Grade 1 bricks from Pittsburgh district laid in cement lime mortar*

FIG. 8.—*Pier B 9, Serial No. 18. Grade 1 bricks from Pittsburgh district laid in lime mortar*

FIG. 6.—Test No. 14. *Grade 2 bricks from Pittsburgh district laid in cement lime mortar*

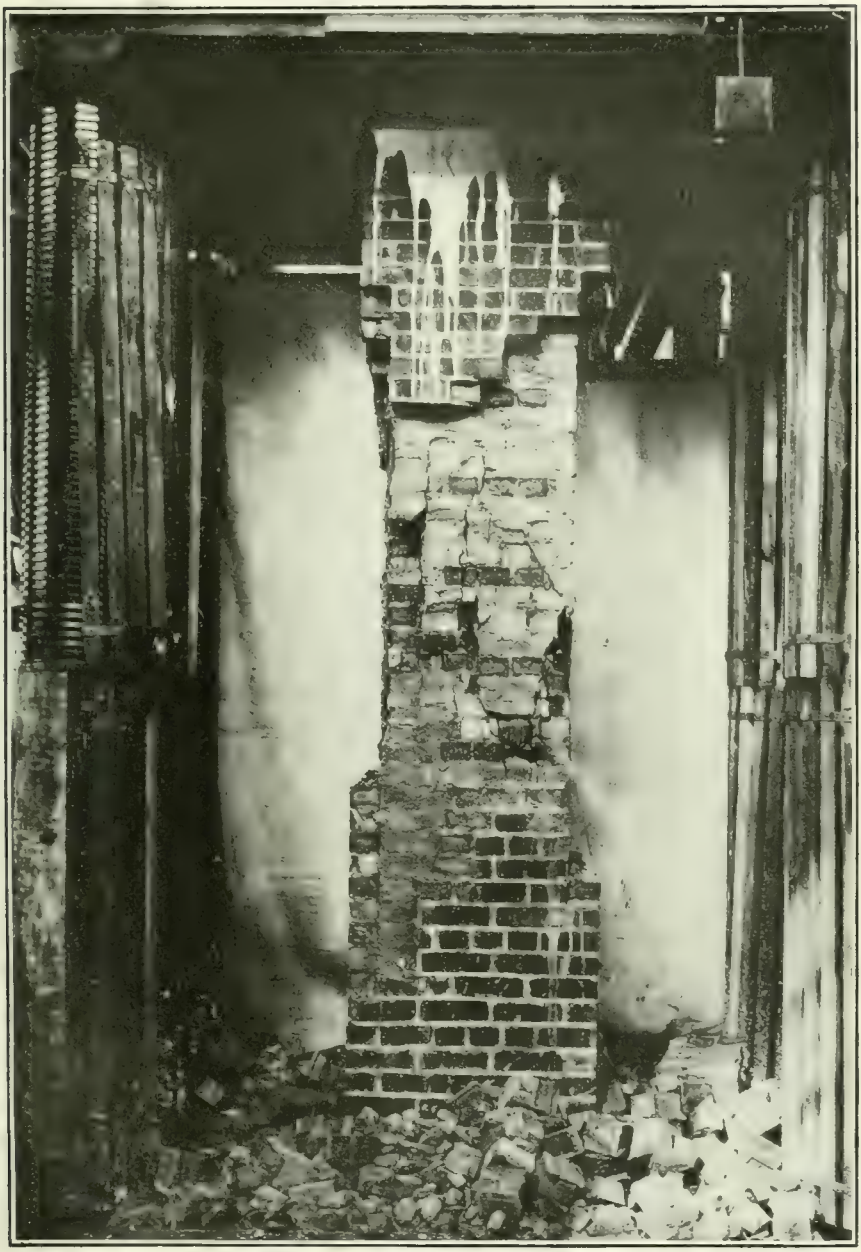


FIG. 10.—*Pier D 42, Serial No. 39. Grade 3 bricks from New York district laid in cement lime mortar*

FIG. 11.—*Pier B 47, Serial No. 25. Grade 1 bricks from New Orleans district laid in cement lime mortar*

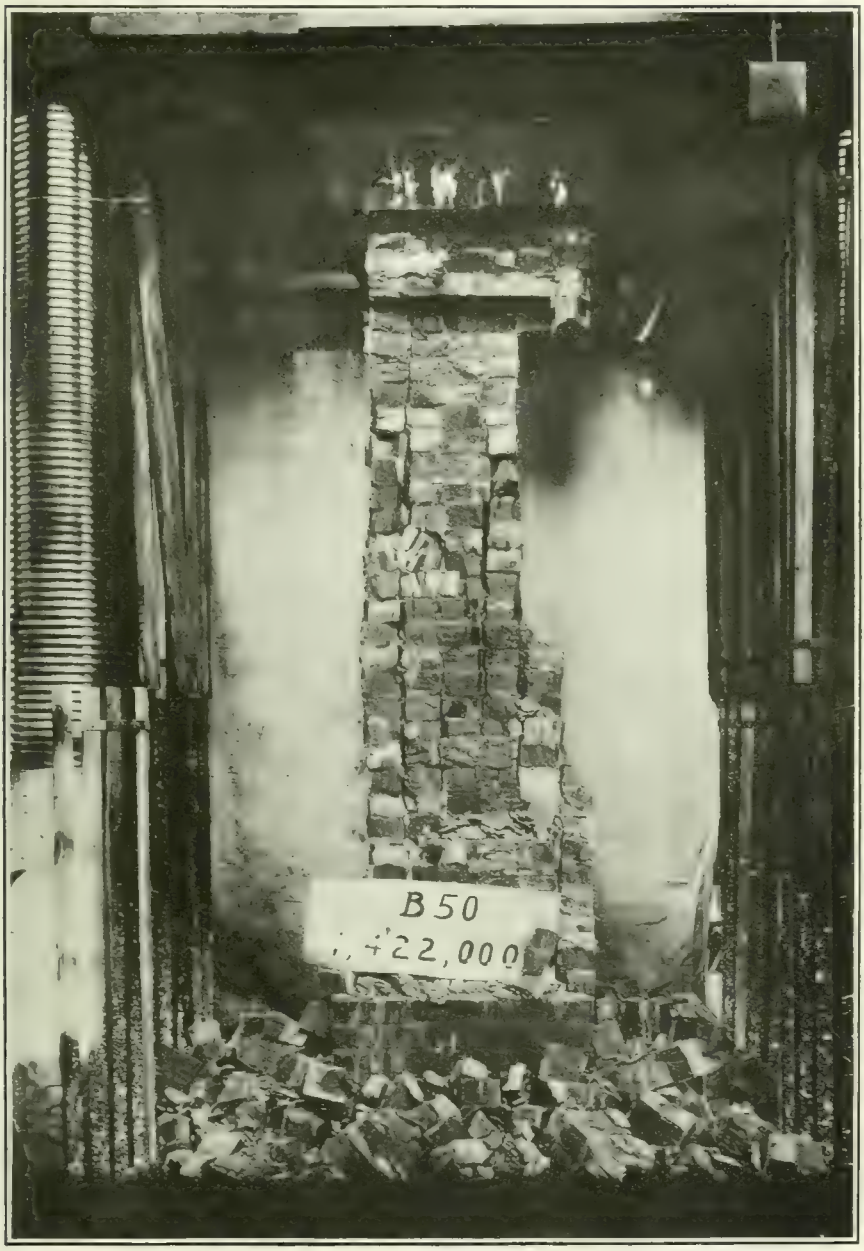


FIG. 12.—*Pier B 50, Serial No. 30. Grade 3 bricks from New Orleans district laid in cement lime mortar.*

Fig. 6.—Pier B 35, Serial No. 41. Grade 1 bricks from Chicago district laid in cement lime mortar

TABLE 8.—Details of Construction and Summary of Test Data. (Piers of Main Investigation)

PITTSBURGH DISTRICT, CEMENT MORTAR

Serial number	Piers: Construction data: Mark on pier	District	Grade of bricks	Bond: Header and stretcher	Courses	Mortar	Height in feet	Piers: Test data: Age in days	Area in square inches	Maximum load in pounds	Maximum load in pounds per square inch	Modulus of elasticity	Bricks: Test data: Average compressive strength, flat	Average compressive strength on edge	Average transverse strength, modulus of rupture	Per cent absorption
1	B13	B	1	1:1	46	1 cement; 3 sand...	10	30	930	2 520 000	2710	2 500 000	11 990	8900	1945	4.08
2	B14	B	1	1:3	46	.....do.....	10	30	930	2 550 000	2740	3 200 000	11 990	8900	1945	4.08
3	B15	B	1	1:6	46	.....do.....	10	30	930	2 697 000	2900	3 200 000	11 990	8900	1945	4.08
4	B17	B	2	1:1	45	.....do.....	10	32	856	1 714 000	2000	2 250 000	7880	6450	1370	7.46
5	B18	B	2	1:3	45	.....do.....	10	32	885	1 834 000	2070	2 000 000	7880	6450	1370	7.46
6	B27	B	2	1:6	44	.....do.....	10	33	946	824 000	870	833 000	2450	2040	675	15.16
7	B1	B	3	1:1	41	.....do.....	10	29	1024	524 000	510	700 000	1659	1350	345	16.28
8	B2	B	3	1:3	41	.....do.....	10	29	1043	580 000	560	733 000	1659	1350	345	16.28
9	B3	B	3	1:6	41	.....do.....	10	32	1024	660 000	650	533 000	1659	1350	345	16.28
PITTSBURGH DISTRICT, CEMENT AND LIME MORTAR																
10	B31	B	1	1:1	45	1 (15 per cent lime, 85 per cent cement); 3 sand.	10	90	841	3 211 000	3800	3 500 000	11 965	10 050	2775	1.28
11	B29	B	1	1:3	45	.....do.....	10	90	841	2 714 000	3220	3 050 000	11 965	10 050	2775	1.28
12	B30	B	1	1:6	45	.....do.....	10	90	841	2 787 000	3300	3 500 000	11 965	10 050	2775	1.28
13	B20	B	2	1:1	45	.....do.....	10	33	908	1 594 000	1760	1 550 000	7880	6450	1370	7.46
14	B28	B	2	1:3	44	.....do.....	10	32	961	838 000	870	750 000	2450	2040	675	15.16
15	B22	B	2	1:6	45	.....do.....	10	30	878	1 545 800	1760	1 625 000	7880	6450	1370	7.46

TABLE 8.- Details of Construction and Summary of Test Data. (Piers of Main Investigation)—Continued

PITTSBURGH DISTRICT, LIME MORTAR

Serial number	Piers: Construction data: Mark on pier	District	Grade of bricks	Bond: Header and stretcher	Courses	Mortar	Height in feet	Piers: Test data: Age in days	Area in square inches	Maximum load in pounds	Maximum load in pounds per square inch	Modulus of elasticity	Bricks: Test data: Average compressive strength, flat	Average compressive strength on edge	Average transverse strength, modulus of rupture	Per cent absorption
16	B7	B	1	1:1	45	1 lime; 6 sand.....	10	120	940	1 360 000	1450	725 000	11 990	8900	1945	4.08
17	B8	B	1	1:3	45	.....do.....	10	120	940	1 197 500	1270	416 700	11 990	8900	1945	4.08
18	B9	B	1	1:6	45	.....do.....	10	120	940	1 280 000	1360	750 000	11 990	8900	1945	4.08
19	B10	B	2	1:1	44	.....do.....	10	120	906	764 000	840	620 000	7880	6450	1370	7.46
20	B11	B	2	1:3	44	.....do.....	10	120	906	804 000	890	687 000	7880	6450	1370	7.46
21	B12	B	2	1:6	44	.....do.....	10	120	900	892 000	990	620 000	7880	6450	1370	7.46
22	B4	B	3	1:1	41	1 lime; 3 sand.....	10	120	1024	215 500	210	300 000	1659	1350	345	16.28
23	B5	B	3	1:3	41	.....do.....	10	120	1024	182 000	178	300 000	1659	1350	345	16.28
24	B6	B	3	1:6	41	.....do.....	10	120	1024	129 000	126	270 000	1659	1350	345	16.28
						NEW ORLEANS DISTRICT, CEMENT AND LIME MORTAR										
25	B47	D	1	1:1	41	1 (15 per cent lime, 85 per cent cement); 3 sand.	10	29	841	1 223 000	1450	1 533 000	7340	4910	733	16.80
26	B48	D	1	1:3	41	.....do.....	10	29	841	1 475 000	1760	1 533 000	7340	4910	733	16.80
27	B52	D	2	1:1	41	.....do.....	10	31	841	1 370 000	1630	1 563 000	6880	5490	893	16.40
28	B53	D	2	1:3	41	.....do.....	10	30	841	1 506 000	1790	1 750 000	6880	5490	893	16.40
29	B49	D	3	1:1	41	.....do.....	10	30	841	1 580 000	1880	1 833 000	6510	5700	1090	17.10
30	B50	D	3	1:3	41	.....do.....	10	29	841	1 422 000	1690	1 583 000	6510	5700	1090	17.10
31	B51	D	3	1:6	41	.....do.....	10	31	841	1 397 000	1660	1 583 000	6510	5700	1090	17.10

NEW YORK DISTRICT, CEMENT AND LIME MORTAR

32	B44	A	1	1:1	45	1 (15 per cent lime, 85 per cent cement); 3 sand.	10	32	791	921 500	1170	875 000	5630	6440	601	16.40
33	B45	A	1	1:3	45	.....do.....	10	32	791	1 033 500	1300	875 000	5630	6440	601	16.40
34	B46	A	1	1:6	45	.....do.....	10	31	791	998 000	1260	875 000	5630	6440	601	16.40
35	B38	A	2	1:1	45	.....do.....	10	30	784	1 001 000	1280	875 000	4430	5449	616	18.60
36	B39	A	2	1:3	45	.....do.....	10	31	791	1 011 000	1280	1 125 000	4430	5449	616	18.60
37	B40	A	2	1:6	45	.....do.....	10	31	791	967 500	1220	817 000	4430	5449	616	18.60
38	B41	A	3	1:1	45	.....do.....	10	31	791	840 500	1070	484 000	2710	2970	497	19.30
39	B42	A	3	1:3	45	.....do.....	10	31	791	840 000	1060	667 000	2710	2970	497	19.30
40	B43	A	3	1:6	45	.....do.....	10	30	791	808 000	1020	600 000	2710	2970	497	19.30

CHICAGO DISTRICT, CEMENT AND LIME MORTAR

41	B35	C	1	1:1	46	1 (15 per cent lime, 85 per cent cement); 3 sand.	10	32	841	706 700	840	833 000	3200	3010	1180	16.20
42	B36	C	1	1:3	46	.....do.....	10	29	812	641 500	790	750 000	3200	3010	1180	16.20
43	B37	C	1	1:6	46	.....d.....	10	30	812	660 500	810	750 000	3200	3010	1180	16.20
44	B32	C	2	1:1	46	.....do.....	10	30	812	606 500	750	833 000	3150	2710	1140	16.20
45	B33	C	2	1:3	46	.....do.....	10	30	812	572 000	700	650 000	3150	2710	1140	16.20
46	B34	C	2	1:6	46	.....do.....	10	30	812	578 500	710	580 000	3150	2710	1140	16.20

TABLE 9.—Details of Construction and Summary of Test Data. (Supplementary Piers)

Serial number	Piers: Construction data: Mark on pier	District	Grade of brick	Type of bond	Mortar	Height in feet	Piers: Test data: Age in days	Area in square inches	Maximum load in pounds	Maximum load in pounds per square inch	Per cent increase or decrease in strength	Bricks: Test data: Average compressive strength, flat	Average compressive strength on edge	Average transverse strength, modulus of rupture	Per cent absorption
47	B23	B	2	1 header, type (a), Fig. 1, to 1 stretcher.	1 (15 per cent lime, 85 per cent cement); to 3 sand.	5	30	915	1 580 000	1730	0	2450	2040	675	15.16
48	B24	B	2	1 header, type (c), Fig. 1, to 1 stretcher.	.....do.....	5	30	915	1 678 000	1840	+6.4	2450	2040	675	15.16
49	B25	B	2	Same as No. 47, with 18 gage No. 2 galvanized-iron wire mesh in every joint.	.....do.....	5	30	924	2 100 000	2270	+31.2	2450	2040	675	15.16
50	B26	B	2	Same as No. 47, with 18 gage No. 2 galvanized-iron wire mesh in every fourth joint.	.....do.....	5	30	930	1 364 000	1470	−15.0	2450	2040	675	15.19

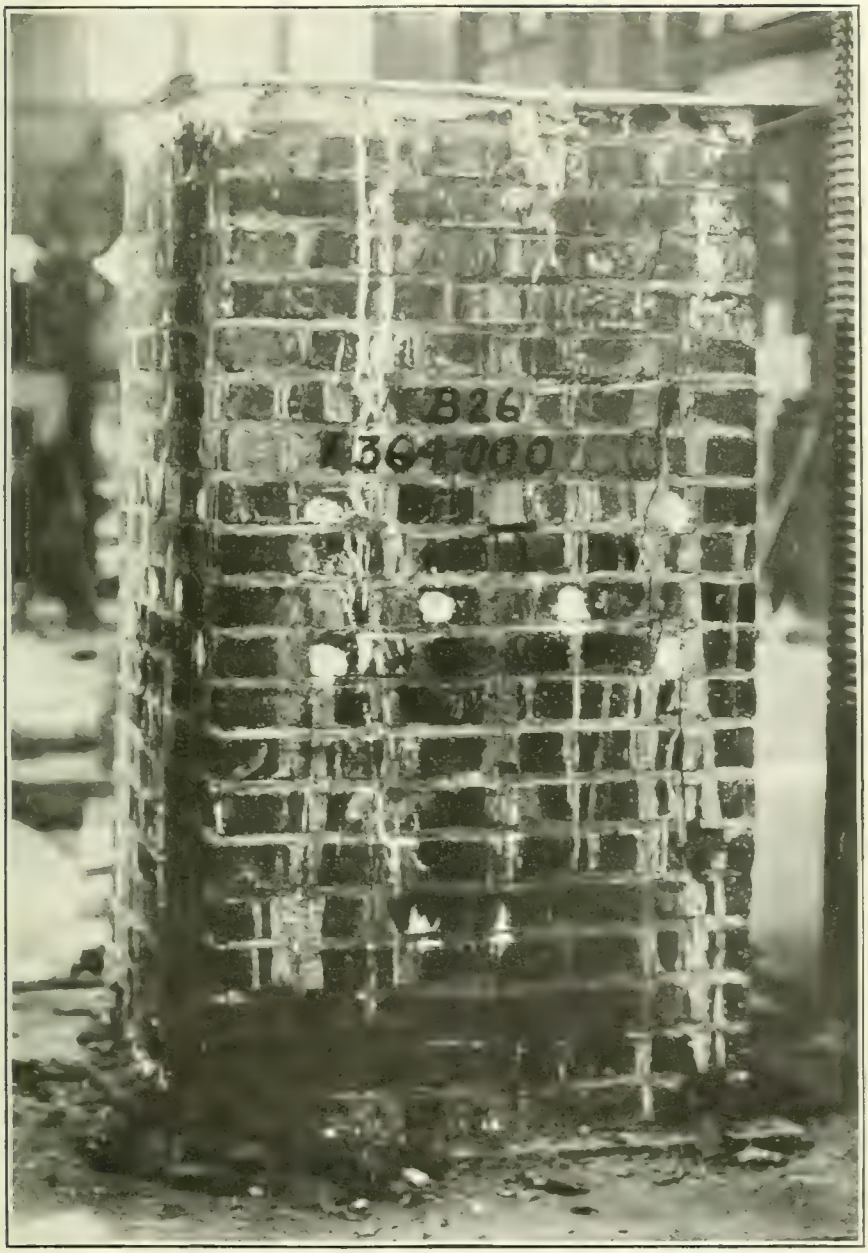


FIG. 14.—*Pier B 26, Serial No. 50. Grade 2 bricks from Pittsburgh district laid in cement lime mortar. Reinforced in every fourth horizontal joint with wire mesh*

TABLE 10.—Representative Test Log of Single Pier. (Pier B 30, Serial No. 12) [a]

Applied load		Readings on north face			Readings on west face		
Pounds per square inch	Pounds	1	2	3	1	2	3
50	42 050	0.0340	0.0260	0.0280	0.0266	0.0292	0.0260
200	168 200	.0344	.0266	.0280	.0274	.0292	.0260
400	336 400	.0350	.0271	.0280	.0282	.0296	.0260
600	504 600	.0354	.0276	.0280	.0284	.0306	.0260
800	672 800	.0361	.0280	.0278	.0290	.0308	.0260
1000	841 000	.0366	.0284	.0278	.0296	.0313	.0258
1400	1 177 400	.0378	.0294	.0276	.0308	.0328	.0258
1800	1 513 800	.0391	.0306	.0273	.0323	.0340	.0256
2200	1 850 200	.0406	.0318	.0268	.0343	.0358	.0250
2600	2 186 000	.0426	.0332	.0265	.0364	.0380	.0244

Applied load		Readings on south face			Readings on east face		
Pounds per square inch	Pounds	1	2	3	1	2	3
50	42 050	0.0351	0.0318	0.0282	0.0270	0.0338	0.0343
200	168 200	.0350	.0318	.0284	.0270	.0341	.0343
400	336 400	.0358	.0322	.0283	.0276	.0346	.0342
600	504 600	.0362	.0328	.0282	.0282	.0252	.0341
800	672 800	.0367	.0333	.0280	.0286	.0357	.0340
1000	841 000	.0373	.0338	.0279	.0292	.0362	.0340
1400	1 177 400	.0383	.0348	.0278	.0302	.0374	.0338
1800	1 513 800	.0394	.0358	.0276	.0309	.0384	.0334
2200	1 850 200	.0307	.0370	.0274	.0320	.0396	.0330
2600	2 186 000	.0325	.0386	.0272	.0336	.0409	.0324

Applied load		Extensometer rod readings			
Pounds per square inch	Pounds	7 northeast	8 northwest	9 southwest	10 southeast
50	42 050	0.0000	0.0000	0.0000	0.0000
200	168 200	.0045	.0088	.0027	.0003
400	336 400	.0107	.0165	.0080	.0065
600	504 600	.0193	.0244	.0140	.0133
800	672 800	.0260	.0312	.0202	.0207
1000	841 000	.0337	.0381	.0263	.0278
1200	1 009 200	.0408	.0460	.0333	.0352
1400	1 177 400	.0480	.0541	.0403	.0420
1600	1 345 600	.0550	.0634	.0478	.0479
1800	1 513 800	.0621	.0729	.0550	.0540
2000	1 682 000	.0703	.0852	.0638	.0603
2200	1 850 200	.0791	.0973	.0731	.0696
2400	2 018 400	.0897	.1086	.0850	.0800
2600	2 186 600	.0905	.1149	.0953	.0900
2800	2 354 800	.0978	.1250	.1083	.1027
3000	2 523 000	.0885	.1265	.1119	.1170
	2 787 000	(b)			

[a] Dimensions of pier, 29 by 29 inches by 10 feet ⅛ inch; bricks, grade 1, District B; number of courses, 45; bond 1, 1 header, 6 stretchers; mortar, 1 (15 per cent lime and 85 per cent cement) to 3 sand; good bearing was obtained over the entire area; pier failed uniformly on all faces; gage length extensometer rods, 11¼ inches; Berry gage length, 8 inches.

[b] Maximum load

TABLE 11.—Representative Test Log of Single Lot of Individual Bricks. (Grade 1 Bricks from District B)

Transverse tests					Transverse tests				
Span in inches l	Width in inches b	Depth in inches d	Load in pounds W	Modulus of rupture $\frac{3Wl}{2bd^2}$	Span in inches l	Width in inches b	Depth in inches d	Load in pounds W	Modulus of rupture $\frac{3Wl}{2bd^2}$
7	3.85	2.30	6300	3250	7	3.85	2.30	3910	2020
7	3.90	2.30	4490	2290	7	3.85	2.30	5300	2740
7	3.85	2.30	5850	3020	7	3.90	2.30	4870	2480
7	3.85	2.30	5800	3000	7	3.90	2.30	5400	2760
7	3.85	2.30	4740	2440	7	3.90	2.30	5360	2740
7	3.85	2.30	5730	2960	7	3.90	2.30	4630	2360
7	3.90	2.30	5870	3000	7	3.90	2.30	5050	2580
7	3.90	2.30	5920	3020	7	3.90	2.30	7230	3690
7	3.90	2.30	5950	3030	7	3.90	2.30	5230	2670
7	3.90	2.30	6075	3100	7	3.90	2.30	4660	2380
					Av....	.....	.....	.....	2775

	Absorption tests			Half brick flat			Half brick on edge		
No.	Dry weight in kilograms	Wet weight in kilograms	Per cent absorption	Dimensions in inches	Load in pounds	Load in pounds per square inch	Dimensions in inches	Load in pounds	Load in pounds per square inch
1	2.876	2.940	2.23	3.6 by 4.3	172 300	11 100	2.3 by 4.2	83 600	8330
2	2.925	2.945	.68	3.9 by 4.2	201 000	12 250	2.3 by 4.3	111 200	11 250
3	2.845	2.866	.74	3.9 by 3.75	180 500	12 350	2.3 by 4.1	103 400	10 900
4	2.845	2.881	1.26	3.9 by 4.0	201 500	12 750	2.3 by 4.3	92 900	9300
5	2.863	2.878	.53	3.9 by 4.15	175 400	10 830	2.3 by 4.2	124 800	12 400
6	2.842	2.864	.77	3.9 by 4.3	219 100	13 050	2.3 by 4.15	79 500	8330
7	2.932	2.975	1.47	3.9 by 4.3	186 000	11 100	2.3 by 4 2	84 300	8720
8	2.866	2.909	1.50	3.9 by 4.2	198 400	12 100	2.3 by 4.25	106 000	10 830
9	2.815	2.874	2.09	3.9 by 4.1	186 400	11 650	2.3 by 3.95	69 000	7600
10	2.885	2.937	1.80	3.9 by 4.05	161 700	10 200	2.3 by 4.4	82 700	8180
11	2.854	2.870	56	3 9 by 3.9	210 200	13 850	2.3 by 4 25	97 000	9920
12	2 920	2 970	1.71	3 9 by 4 4	211 800	12 350	2.3 by 4.05	138 500	14 870
Av.	.....	.....	1.28	.....	.....	11 965	.....	.....	10 050

2. DERIVED DATA

(A) Relation of Transverse Strength of Bricks to Compressive Strength of Piers. - For the most part, efforts to establish ..... relations between the compressive strength of the bricks and the ultimate strength of the piers have met with small success except in cases previously referred to where the conditions pertaining to the erection of the test piers were ideal. Observations recorded in the first few tests of the present investi-

gation pointed to certain characteristics of failure indicating that the transverse strength of the bricks might bear a close relation to the ultimate strength of the piers. For this reason the transverse strength was determined on each lot of bricks and the

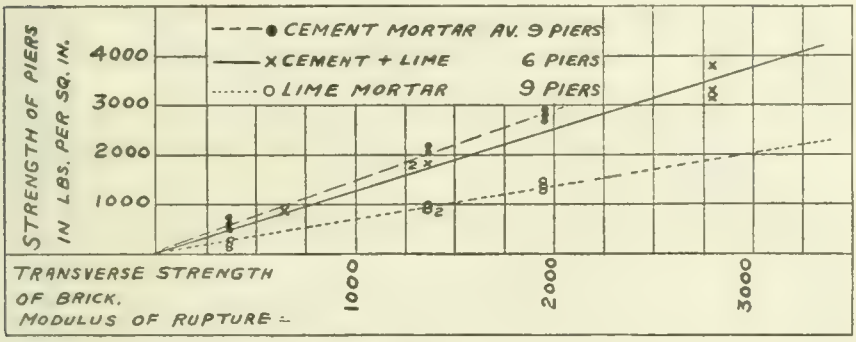


FIG. 15.—*Showing relation of transverse strength of individual bricks to compressive strength of piers, representing three grades of brick and three mortars from Pittsburgh district*

average unit transverse strength or modulus of rupture obtained. These averages have been plotted as abscissas and the unit loads sustained by the piers as ordinates and are shown in Figs. 15 and 16. Fig. 15 includes only piers of the Pittsburgh district repre-

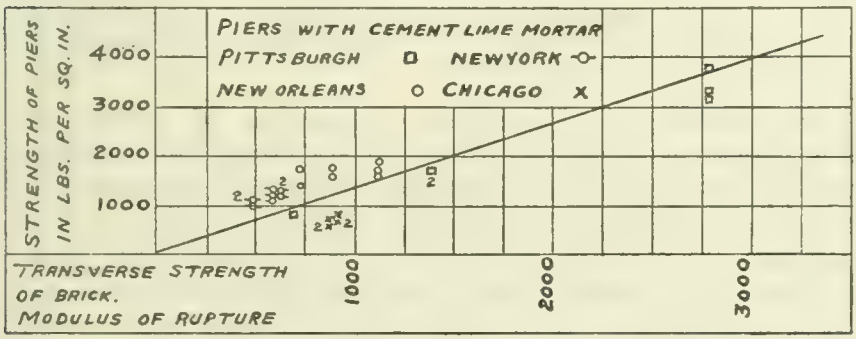


FIG. 16.—*Showing relation of transverse strength of individual bricks to compressive strength of piers, representing three grades of brick from four districts. All piers laid in cement-lime mortar*

senting three grades of mortars, while Fig. 16 includes piers from all districts laid in cement-lime mortar. In both cases it is shown that the transverse strength of the bricks is proportional to the compressive strength of the piers. For use in computing the

strength of brick piers the empirical formula $P = K\,R$ is derived from these plots. In this formula

P = the compressive strength of the pier in pounds per sq. in.,

R = the modulus of rupture of the bricks,

K is a constant depending upon the kind of mortar used.

The values of K for mortars included in these tests are as follows:

For 1 part Portland cement to 3 parts sand, $K = 1.45$,

For 1 part (15 per cent lime 85 per cent cement) to 3 parts sand, $K = 1.25$,

For 1 part hydrated lime to 6 parts sand, $K = 0.65$.

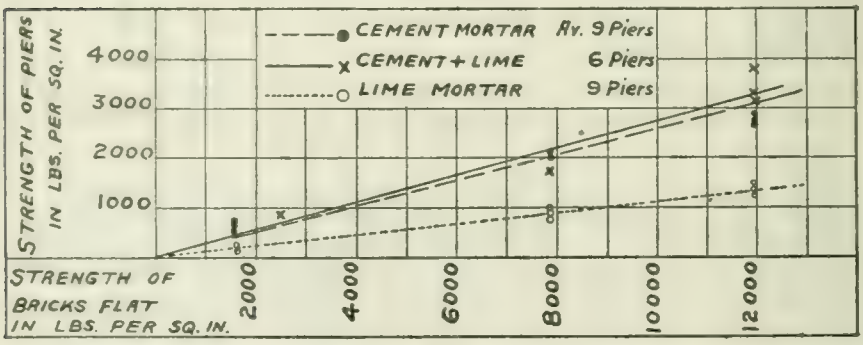


FIG. 17.—*Relation of compressive strength of single bricks (flat) to compressive strength of piers, representing three grades of brick from four districts. All bricks laid in cement-lime mortar*

(B) RELATION OF COMPRESSIVE STRENGTH OF BRICKS TO COMPRESSIVE STRENGTH OF PIERS.—The average unit compressive strength of the half bricks tested flat and on edge have been plotted in the same manner as the modulus of rupture and are shown in Figs. 17 to 20, inclusive. Figs. 17 and 19 are representative of the three different grades of mortar, while Figs. 18 and 20 include piers of all districts laid in cement-lime mortar. In these plots it is shown that the compressive strengths of the bricks is proportional to the ultimate compressive strength of the piers and the formula

$P = K\,p$ is derived, in which

P = the unit compressive strength of the pier,

p = the unit compressive strength of the bricks,

K is a constant, depending upon the grade of mortar.

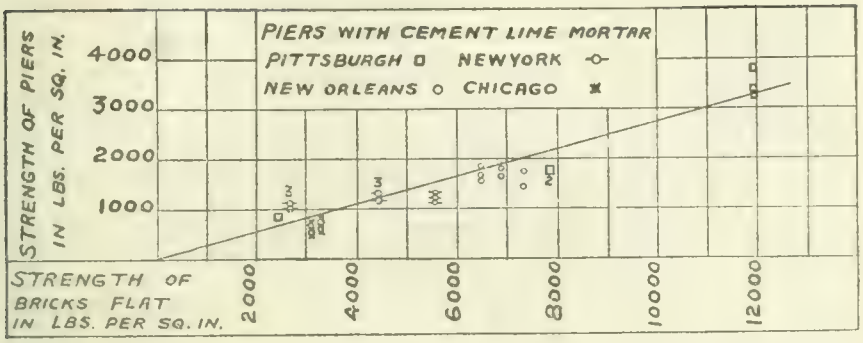


FIG. 18.—*Relation of compressive strength of single bricks (flat) to compressive strength of piers, representing three grades of brick and three mortars from Pittsburgh district*

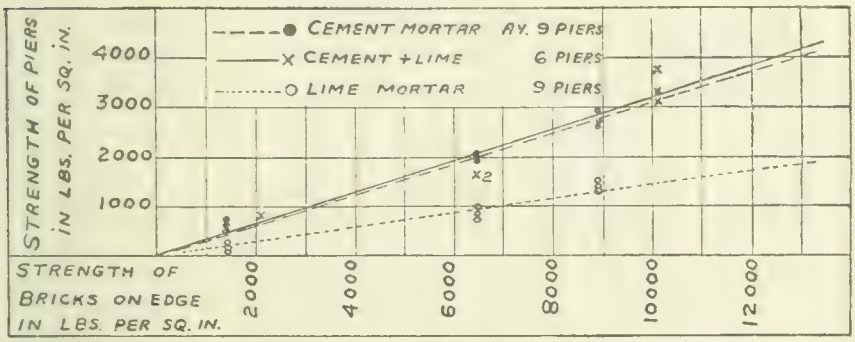


FIG. 19.—*Relation of compressive strength of single bricks (on edge) to compressive strength of piers, representing three grades of brick and three mortars from Pittsburgh district*

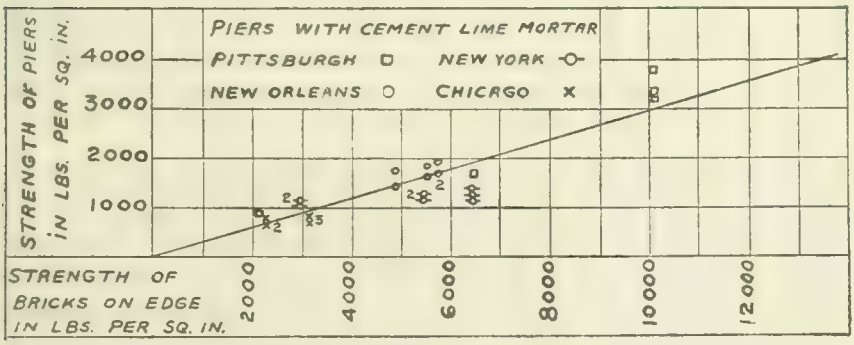


FIG. 20.—*Relation of compressive strength of single bricks (on edge) to compressive strength of piers, representing three grades of brick from four districts. All piers laid in cement-lime mortar*

The following table gives the value of K for the grade of mortar employed:

Bricks tested	Mortar	Value of K
Flat	1 cement to 3 sand by weight	0.27
	1 (15 per cent lime, 85 per cent cement) to 3 sand by weight	.26
	1 lime hydrate to 6 sand by weight	.11
On edge	1 cement to 3 sand by weight	.32
	1 (15 per cent lime, 85 per cent cement) to 3 sand by weight	.30
	1 lime hydrate to 6 sand weight	.14

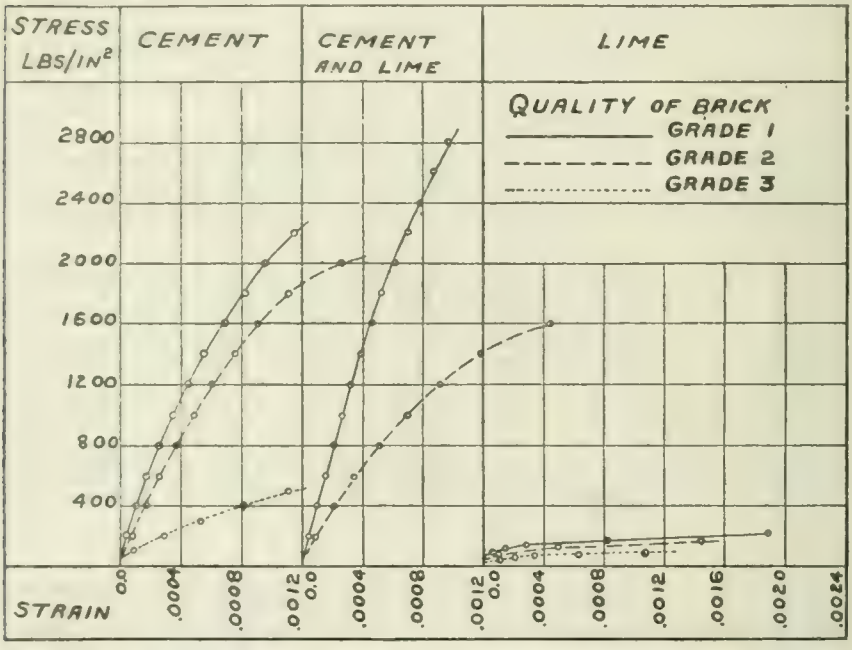


FIG. 21.—*Stress strain curves showing relative degree of compressibility of the various grades of brick laid in different kinds of mortar*

(C) MODULUS OF ELASTICITY.—The modulus of elasticity of the piers has been determined from the stress-strain curves obtained from the full-length compression readings. Representative stress-strain curves are shown in Fig. 21. This figure shows the relative stiffness of piers built of different grades of bricks and mortars. There is very little difference in the modulus of elasticity for piers of cement mortar and those of cement-lime mortar in which bricks of the same grade were used. There is a big difference, however, in the modulus of piers of these mortars and those of pure lime mortar. It may also be seen that

in the case of cement and cement-lime mortars, the piers of low-grade bricks have a considerably lower modulus than those of the higher grades, while very little difference is apparent in the case of pure lime mortar. In Fig. 22 it is shown that in the cement and cement-line mortar piers the modulus of elasticity is proportional to the compressive strength of the pier.

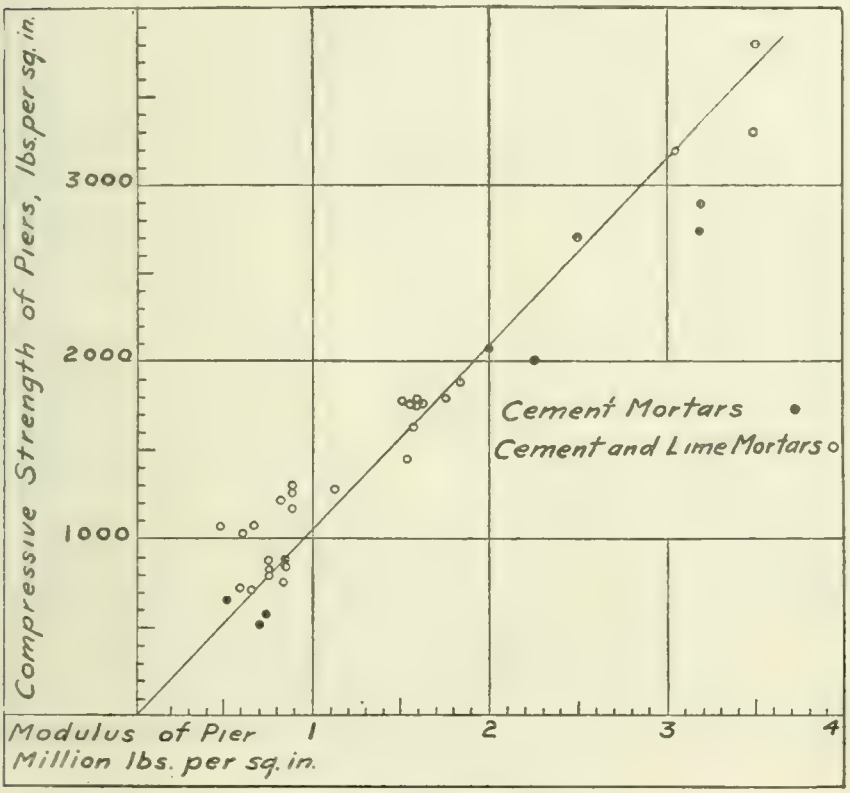


FIG. 22.—*Showing relation of modulus of elasticity to compressive strength of piers*

(D) LATERAL STRAINS.—The type of failure peculiar to brick piers indicates an outward flow of the materials with a consequent tension failure of the individual bricks. Prof. J. B. Johnson [5] implies that for this reason the tensile strength of the brick is a very important quality. Such a condition can be possible only if there is a tendency of the mortar in the horizontal joints to flow faster than the bricks adjacent to the joints. In the present investigation strain measurements were made in the horizontal joints and on the adjacent courses of

[5] Johnson's Materials of Construction, 4th ed., 1907, p. 654.

brick. The results obtained on two representative piers are shown in Figs. 23 and 24. These diagrams show that the strain is sometimes greater in the joints and sometimes greater in courses adjacent to the joints. When the measurements on the four faces of the pier are averaged it is shown that there is no appreciable difference between the strain in the joints and the strain in the adjacent courses.

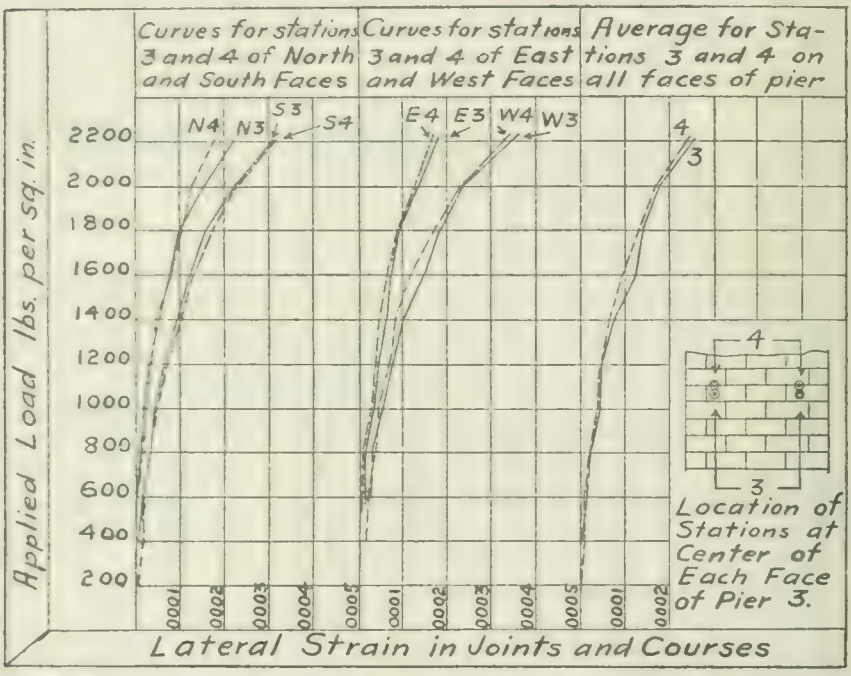


FIG. 23.—*Showing relative strain in horizontal joints and adjacent courses on pier of grade I bricks laid in cement mortar*

VI. GENERAL DISCUSSION

1. TYPE OF FAILURE

The type of failure observed in this investigation is in agreement with previous studies of the compressive strength of brick masonry. It may be said, therefore, that failure by vertical cleavage is common to all concentrically loaded brick piers. The first indication of failure appears at about 75 per cent of the ultimate compressive strength of the pier and is indicated by the appearance of hair-size vertical cracks in the individual bricks. These cracks usually start at or quite near the center of the bricks where the bricks break joints with the adjacent courses. The

cracks gradually extend to other courses, and finally, becoming confluent, they extend almost the entire length of the pier. Soon thereafter final failure occurs, accompanied by a spawling off of the outer rings of bricks. In the case of piers of grade 1 bricks laid in lime mortar, sharp reports are heard as the cracks begin to appear, and they increase in frequency with additional load. These reports are peculiar to the failure of bricks by flexure as distinguished from sounds accompanying compression failure. Except in cases of piers laid in mortar of equal or greater strength

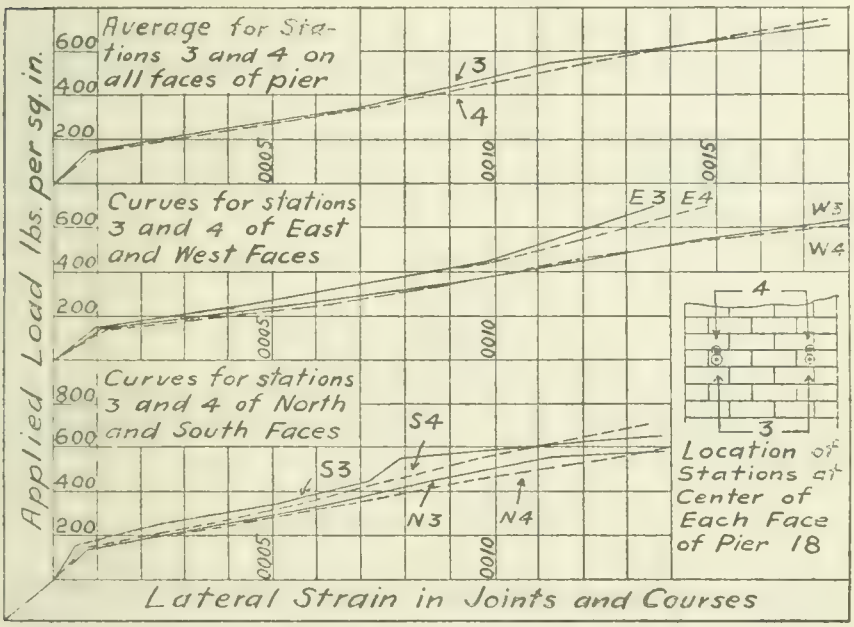


FIG. 24.—*Showing relative strain in horizontal joints and adjacent courses on pier of grade 1 bricks laid in lime mortar*

than the bricks, it is seen after failure that the bricks are broken in two or more pieces and very little crushing of the bricks is apparent. All indications point to a transverse failure of the individual bricks as the cause of incipient failure of the pier. In general, conditions are more favorable to this type of failure of the bricks than to the failure by compression. In most cases the mortar has less stiffness than the brick. Unless all joints are of equal thickness this will cause unequal loading of the individual bricks as the pier is compressed. The same thing will occur when the joints are of equal thickness, but if at the same time the mortar is not distributed over the entire bearing surface of the brick.

Irregularity in the shape of the bricks will result in varying thickness of mortar on their surfaces.

The transverse strength of a single brick varies as the breadth, the depth squared, and inversely as the distance between supports, or

$P = \frac{kbd^2}{l}$. In this equation

P = the breaking load of the brick, in pounds.
b = breadth, in inches.
d = depth in inches.
l = span in inches.
k is an experimental constant depending upon the grade of brick.

As an illustration of how the transverse strength of a single brick varies with the depth, the following values are substituted in the above formula:

k = variable for different grades of brick = 1000,
b = breadth of brick = 4 inches,
d = depth of brick = 2 inches,
l = distance between supports = 7 inches.

Then for a brick in a flatwise position supported at the ends and loaded at the center,

$$P = \frac{1000 \times 4 \times 4}{7} = 2287 \text{ pounds.}$$

When the brick is tested in the edgewise position the breadth is decreased from 4 inches to 2 inches and the depth is increased from 2 inches to 4 inches, then

$$P = \frac{1000 \times 2 \times 16}{7} = 4574 \text{ pounds.}$$

This is twice the strength developed in the flatwise position. It follows, then, that any method of construction which would increase the depth of the bricks or component parts of the pier should increase the strength of the pier. In the Watertown Arsenal tests this was done by laying the bricks on edge, also by breaking joints every sixth course when laid flatwise and every third course when laid on edge. In these tests face brick were laid in 1:1 cement mortar. The following table shows the gain in strength due to the method of construction:

Method of laying bricks	Percentage gain in strength
Bricks flat:	
Joints broken every course	0
Joints broken every sixth course	7.6
Bricks on edge:	
Joints broken every course	43.8
Joints broken every third course	57.3

It is quite probable that the gain in strength due to the method of laying would be greater for softer mortars. That there is a close relation between the transverse strength of the bricks and the ultimate compressive strength of the piers is shown in Fig. 5. The lines connecting the points in this diagram indicate by direction how the strength of the pier follows the strengths of the individual bricks. It is also significant that in the empirical formula given by H. Kreuger a different constant is used for different thickness of brick. He also states that the piers of thinner bricks develop less strength than those of thicker bricks.

2. EFFECT OF QUALITY OF BRICKS

The quality of bricks is obviously a very important factor in the strength of brick masonry. While a high compressive strength of the brick contributes to the ultimate strength of the pier, it is shown also that other qualities of the brick are important in their effect on the compressive strength of masonry. Tests show that the transverse strength of the brick bears a close relation to the strength of the pier. In this respect regularity in shape is essential, since if the bricks are irregular they can not be evenly bedded in the mortar, and this will cause the joints to be thicker than is desirable. Also with bricks of equal compressive strength but of varying depth, the piers built of bricks having the greatest depth will develop higher compressive strengths. The degree of absorption is important in its effect on the strength of the mortar. Very porous brick must be thoroughly "wet down" to prevent absorption of the water in the mortar, which in the case of the richer cement mortars is necessary to the proper hydration of the cement. That the quality of brick with respect to strength is of great importance is seen in Figs. 15 to 20, inclusive, which show that the strength of the piers is proportional to the strengths of the bricks used in their construction.

3. EFFECT OF MORTARS

The importance of proper proportioning of the mortar to be used in brickwork can not be emphasized too strongly. A pure Portland cement mortar, when tested in the form of cubes or cylinders, develops a higher compressive strength than lime or combination cement and lime mortars. Tests show, however, that piers laid in pure Portland cement mortar do not always develop the highest strengths when compared with piers of combination cement and lime mortar. In the present investigation it is seen that there is practically no difference in strength in the piers of pure Portland cement mortar and those of the cement-lime mortar. The cement-lime mortar used was proportioned by weight, 15 per cent of the cement being replaced by hydrated lime. The amount of cement replaced by lime is, therefore, about 35 per cent by volume. The tests made by Macgregor show that in a 1:3 cement and sand mortar 50 per cent of the cement may be replaced by lime without decreasing the strength of 8-inch piers. It is believed, however, that the strengths developed in these tests is higher than would be realized with the same mortar on piers of greater cross sectional dimensions. The greatest advantage in the replacing of a part of the cement with hydrated lime is in the easier working qualities of the cement-lime mortar. Since the cement-lime mortar is more plastic than the pure Portland cement mortar, the bricks may be laid with greater ease and a more nearly uniform bedding of the bricks obtained. That a pure hydrated lime mortar is inefficient in masonry where a high compressive strength is desired is shown conclusively in these tests. The piers of pure lime mortar, although aged four months as compared with one month for the piers of other mortars, showed when broken an almost entire lack of carbonation of the mortar on the interior. It is seen also in Fig. 21 that the cement and cement-lime mortar piers will compress only about one-fourth to one-half as much for the same load as those of the pure lime mortar. A low degree of compressibility is, of course, desirable for masonry in foundations or other parts of a building subjected to compression stresses.

4. EFFECT OF BOND

The opinion prevails that the tying in of the masonry with header courses helps to strengthen the pier against bulging action, thereby increasing the strength in proportion to the number of headers used. Results obtained in the present investigation,

however, show that variations in the number of header courses used do not have a positive effect on the compressive strength of the pier. A comparison of the strength of the piers with respect to the number of header courses is given in the following tabulation:

11 GROUPS OF 3 PIERS EACH

Compressive strength of pier	Number of header courses to stretcher courses		
	1:1	1:3	1:6
Highest	7	1	3
Intermediate		6	5
Lowest	4	4	3

3 GROUPS OF 2 PIERS EACH

Highest		3	
Intermediate			
Lowest	3		

The header course used in these tests, Fig. 1 (*a*), is believed to be the only practicable one which the size of the pier will permit. However, the earlier piers of the Pittsburgh district indicated a decrease in strength of the piers having the greatest number of header courses. It was thought that this effect was probably due to the presence of so many broken or quarter bricks in the corners of the header course. This led to a special study of bond, the results of which are given in Table 2. In these tests pier 48 was built, using header (*c*), Fig. 1. Although this header has fewer broken bricks, it necessitates a special arrangement of the adjacent stretcher courses in order to properly break joints. While the strength of this pier is 6 per cent higher than that of pier 47, in which header (*a*), Fig. 1, was used, the result is not conclusive, since in two piers of exactly the same construction large variations in strength may be expected. In view of the tendency of the individual bricks to fail by flexure, it is believed that the bricks of the header courses are broken through at the joints before the pier has reached that point of failure where these bricks would prevent bulging of the outer rings. For this reason very little increase in strength may be expected by the introduction of header courses. The results obtained on the piers reinforced with wire mesh are in close agreement with those made at Cornell University and indicate that while wire mesh in every horizontal joint increases the strength of the pier, the gain is slight when mesh is used only in every third or fourth course.

VII. SUMMARY

The following conclusions are based on the results of the present investigation and a study of previous tests referred to in Section II:

1. The primary failure of brick piers is caused by a transverse failure of the individual bricks.

2. The ultimate strength of the pier may be increased by any method of construction which will increase the depth of the component parts of the pier. This may be done by (1) laying the bricks on edge instead of flat, (2) breaking joints every few courses instead of every course, or (3) using bricks of more than ordinary thickness.

3. The strength of the pier may be increased by the introduction of wire mesh in all horizontal joints. The increase is slight, however, unless the mesh is used in every joint.

4. Varying the number of header courses used does not appreciably affect the ultimate strength of the pier.

5. The mortar joints should be made as thin as possible. They should be of uniform thickness. For this reason regularity in shape of the bricks is essential.

6. The ultimate strength of brick piers is proportional to the compressive and transverse strength of the bricks used in their construction.

7. The kind of mortar used is important in its effect on the strength of brick masonry. A pure lime mortar is inefficient when a high compressive strength is desired. In a mortar of 1 part Portland cement to 3 parts sand, 25 per cent by volume of the cement may be replaced by hydrated lime without appreciably affecting the strength of brick piers. Higher percentages of lime up to 50 per cent by volume may be used on piers of small cross sectional dimensions.

8. The following empirical formulas for use in computing the strength of brick piers are derived from the tests of the present investigation:

$$(1)\quad P = K\,p$$

$$(2)\quad P = K\,R,\ \text{where}$$

P = the ultimate unit compressive strength of the pier,

p = the unit compressive strength of the single bricks,

R = the unit transverse strength or modulus of rupture of the single bricks,

K is a constant depending upon the grade of mortar used, and for which the following values are given.

Mortar	Values to be determined by tests of individual bricks	Value of K
1 (15 per cent lime; 85 per cent cement) to 3 parts sand, by weight.	Unit compressive strength flat $= f$	0.26
	Unit compressive strength on edge $= f$	.30
	Modulus of rupture from transverse test $= R$	1.25
1 part lime to 6 parts sand	Unit compressive strength flat $= f$	.11
	Unit compressive strength on edge $= f$	.14
	Modulus of rupture from transverse test $= R$	.65
1 part cement to 3 parts sand	Unit compressive strength flat $= f$	.27
	Unit compressive strength on edge $= f$	.32
	Modulus of rupture from transverse test $= R$	1.45

Much valuable assistance has been rendered the author throughout the investigation, also in the preparation of this report, and it is desired especially to mention Prof. A. V. Bleininger and J. H. Griffith, of the Bureau of Standards, and Edward Orton, jr., chairman of the technical committee of the National Brick Manufacturers' Association.

WASHINGTON, December 12, 1917.

DEPARTMENT OF COMMERCE

TECHNOLOGIC PAPERS

OF THE

BUREAU OF STANDARDS

S. W. STRATTON, DIRECTOR

No. 112

STANDARDIZATION OF THE SAYBOLT UNIVERSAL VISCOSIMETER

BY

WINSLOW H. HERSCHEL, Associate Physicist
Bureau of Standards

[2d Edition]
ISSUED APRIL 30, 1919

PRICE, 10 CENTS
Sold only by the Superintendent of Documents, Government Printing Office, Washington, D. C.

WASHINGTON
GOVERNMENT PRINTING OFFICE
1919

STANDARDIZATION OF THE SAYBOLT UNIVERSAL VISCOSIMETER

By Winslow H. Herschel

CONTENTS

	Page
1. Introduction	3
2. The need of standardization	4
3. The standard dimensions of the Engler viscosimeter	4
4. The measurement of the dimensions of the Saybolt Universal viscosimeter	5
(*a*) Meissner's determinations	6
(*b*) Measurements by the Bureau of Standards	6
5. The effect of variations in dimensions of minor importance	8
6. Equation for determining viscosity from dimensions of efflux instrument and the time of discharge	9
7. Higgins's method for determining the relation between kinematic viscosity and time of discharge	10
8. A method of finding the constant A in equation (4)	14
9. Determination of the constant B by Higgins's method	16
10. Determination of the Couette correction and the kinetic energy correction by Herschel's method	16
11. Equations for instruments not of normal dimensions	19
12. Applications of the new equations	20
13. The Redwood viscosimeter	21
14. Conclusion	22
Appendix	23
1. The viscosity of glycerol solutions	23
2. The viscosity of castor oil	23

1. INTRODUCTION

The Saybolt Universal viscosimeter has always been at a disadvantage, as compared with the Engler instrument, because it had never been standardized with respect to its principal dimensions. To overcome this difficulty the Bureau of Standards entered into negotiations with George M. Saybolt, and he agreed to accept certain instruments as standard.

In a previous paper the method was described for determining an equation showing the relation between readings of certain Saybolt Universal viscosimeters and the absolute viscosity. The object of the present investigation has been to determine a similar equation for instruments of standard dimensions. As the methods employed have been mainly the same as before,

frequent references to Technologic Paper No. 100 will be necessary, although it will, in general, be assumed that the reader is familiar with the efflux method of determining viscosity, as described in that paper.

2. THE NEED OF STANDARDIZATION

In calculations involving viscosity such units as Saybolt seconds and Engler degrees can not be used. It is necessary to express viscosity by an absolute unit whose value is independent of the instrument by which it is obtained. To completely standardize a viscosimeter, therefore, it is necessary to adopt normal dimensions, with allowable tolerances, and to determine an equation for converting viscosimeter readings into absolute viscosity.

For measuring the viscosity of oils the two most commonly used efflux viscosimeters are the Saybolt Universal and the Engler. In both instruments there is a cylindrical container for the liquid whose viscosity is to be measured, an outlet tube at the bottom of the container, and a bath surrounding the container to control the temperature. The instruments differ in the dimensions of the various parts and in the method of starting the flow. The essential dimensions of the Saybolt instrument have been published by Gill[1] and by Meissner,[2] but they are not in good agreement, so that all equations previously determined must be regarded as applying to certain instruments, but not to instruments of standard dimensions.

3. THE STANDARD DIMENSIONS OF THE ENGLER VISCOSIMETER

The Engler instrument has been standardized with respect to its principal dimensions, although, as has been indicated elsewhere,[3] the much-used Ubbelohde equation is inaccurate. As the methods of standardizing the Engler viscosimeter apply for the most part to the Saybolt instrument also, it will be of interest to consider them briefly. The Engler instrument was originally described by its designer,[4] and on April 1, 1907, the principal German testing laboratories adopted the normal dimensions and tolerances as given in Table 1.[5]

---

[1] A. H. Gill, Oil Analysis, p. 29; 1913.

[2] W. Meissner, Chemische Revue über die Fett- und Harz-Industrie, **19**, p. 9; 1912.

[3] Winslow H. Herschel, Technologic Paper No. 100, Bureau of Standards, p. 27; 1917. (This paper will be referred to subsequently simply as T. P. 100.)

[4] C. Engler, Zeitschrift für angewandte Chemie, **5**, p. 725; 1892.

[5] See Engler-Hofer, Das Erdol, **4**, p. 99, 1913; Chem. Ztg., **31**, p. 447, 1907; Chem. Revue über die Fett- und Harz-Industrie, **14**, p. 118, 1907; Holde-Mueller, The Examination of Hydrocarbon Oils, p. 105, 1915. (The obsolete reference to 240 cc may still be found in many books.)

TABLE 1.—Standard Dimensions of Engler Viscosimeter

Specified distances	Normal dimensions	Tolerance
	Cm.	Cm.
Length of outlet tube, l	2.0	±0.01
Inside diameter of outlet tube, at upper end, d_1	.29	± .002
Inside diameter of outlet tube, at lower end, d_2	.28	± .002
Outside diameter of outlet tube, d_3	.45	± .02
Length of outlet tube, projecting below bottom of the bath, l_1	.30	± .03
Inside diameter of container, D	10.6	± .10
Depth of cylindrical part of container below the gage points, h_3	2.5	± .10
Height of the gage points above lower end of outlet tube=initial head, h_1	5.2	± .05

It was originally directed to measure out 240 cm^3 of liquid in a flask and to pour into the container, but it is evident that the level of the liquid at the start, rather than the volume in the container, is the factor which controls the pressure which in turn produces the flow. For the volume of the dished part of the container—that is, the capacity of the container below the gage points—in excess of the 200 cm^3 to be withdrawn, can have no effect upon the head causing flow. Moreover, since the contour of the bottom of the container is not specified, it might be expected to differ in different instruments and cause a consequent variation in the volume contained in the dished part.

The directions finally adopted are to pour in an excess of liquid so as to cover the gage points, and then adjust the height of the liquid to the level of the gage points by removing the excess with a pipette. The time of discharge for water at 20° C (68° F), usually called the water rate, must lie between 50 and 52 seconds. Meissner [6] says that it is 50.94 seconds for an instrument of normal dimensions.

4. THE MEASUREMENT OF THE DIMENSIONS OF THE SAYBOLT UNIVERSAL VISCOSIMETER

It has long been realized that the Saybolt instrument would remain at a great disadvantage in competition with the Engler, until it was standardized by the adoption of normal dimensions and suitable tolerances similar to those given in Table 1 for the Engler instrument. As a result of negotiations between George M. Saybolt and this Bureau, Mr. Saybolt sent four viscosimeters, Nos. 108 to 111, to be measured and to serve as a basis for the adoption of normal dimensions and tolerances.

[6] W. Meissner, Chem. Revue über die Fett- und Harz-Industrie, 17, p. 202; 1910.

As the time of discharge, which is used as a measure of the viscosity, varies very rapidly with the diameter of the outlet tube, it was necessary that this dimension should be measured with great accuracy.

(*a*) MEISSNER'S DETERMINATIONS.—Meissner's methods of measurement are suggestive and will be briefly described. The mean diameter of the outlet tube was determined as follows:

> First, cylindrical rods were turned to exactly fit the tube. Secondly, the diameter was measured at the upper and lower ends by means of a special plug gage of circular section, with a 1 : 40 taper. The mean was taken of these three measurements. The value of d_m is nevertheless subject to considerable uncertainty since the workmanship of the tube * * * is not precise. * * * d varied between 1.77 and 1.79 mm.

The length of the outlet tube, l, and the initial head, h_1, were determined with the help of a rod turned small enough to pass easily through the outlet tube as far as a shoulder. To determine l the rod was put through the tube from above, and the length of rod projecting below the bottom of the tube was measured with a vernier depth gage. To find h_1 the rod was put through the tube from below, and the depth gage was used to measure the distance from the upper end of the rod to the overflow rim which fixes the initial level of the liquid.

(*b*) MEASUREMENTS BY THE BUREAU OF STANDARDS.—The dimensions of Saybolt viscosimeters Nos. 108 to 111 were determined without the use of special gages. The inside diameters of the outlet tubes were measured with a micrometer microscope. In finding the diameter at the middle of the tubes it was found convenient to focus on the points of inside calipers made by bending a wire in the form of a V or Y, although the calipers could often be dispensed with by focusing on particles of dust. The length of tube was found by subtracting the distance from the overflow rim to the upper end of the outlet tube from the overall distance from the rim to the lower end of the tube. The final results of the measurements of these viscosimeters are given in Table 2. The symbols for the dimensions are the same as in Table 1.

TABLE 2.—Dimensions of Saybolt Universal Viscosimeters

Dimension	Values as measured by Bureau of Standards			
	No. 108	No. 109	No. 110	No. 111
l ..... centimeters..	1.217	1.233	1.235	1.230
d average ..... do....	.1781	.1764	.1759	.1770
d_3 outside ..... do....	.30	.31	.30	.28
D ..... do....	2.975	2.972	2.974	2.974
h_1 ..... do....	12.448	12.542	12.520	12.551
h_2 ..... do....	3.816	3.893	3.882	3.913
h average ..... do....	7.300	7.393	7.377	7.411
Capacity of container ..... cubic centimeters..	69.4	69.5	69.8	69.6

Table 3 gives the normal dimensions and allowable variations proposed by the Bureau of Standards and accepted by Mr. Saybolt on October 1, 1917. It will be seen that the normal dimensions are practically the same as the average of corresponding dimensions in Table 2. The tolerances were determined by a compromise between two considerations. Too large a tolerance would cause too great a variation in efflux time between different instruments, while too small a tolerance would unnecessarily increase the cost of manufacture in attempting to obtain a degree of accuracy not required in technical work. For precise scientific work, some form of viscosimeter should be used which is provided with an outlet tube of much greater length as compared with its diameter.[7]

TABLE 3.—Dimensions of the Standard Saybolt Universal Viscosimeter

Dimension	Minimum	Normal	Maximum
	Cm	Cm	Cm
Diameter of outlet tube, d	0.1750	0.1765	0.1780
Length of outlet tube, l	1.215	1.225	1.235
Outer diameter of outlet tube, at lower end, d_3	.28	.30	.32
Height of overflow rim above bottom of outlet tube, h_1	12.40	12.50	12.60
Diameter of container, D	2.955	2.975	2.995
Average head, h (calculated)	7.16	7.36	7.56

[7] T. P. 100, p. 12.

5. THE EFFECT OF VARIATIONS IN DIMENSIONS OF MINOR IMPORTANCE

The Saybolt viscosimeter is so constructed that the lower end of the outlet tube is about 1.0 cm above the bottom of the bath, the exact distance depending upon the thickness of washers in compression. This distance, therefore, can not be specified, as with the Engler instrument where the bath and container are rigidly soldered together. If, in the Engler instrument, l_1 were too short, the liquid might spread out on the bottom of the bath, thus increasing the effect of surface tension, while if l_1 were too long, the temperature of the liquid in the outlet tube might be perceptibly lower than the temperature of the liquid in the container. These difficulties are avoided by the design of the Saybolt instrument.

Of the dimensions in Table 3, the least important is the outer diameter of the outlet tube. This determines the resistance to flow due to surface tension, which must be considered because the discharge is not submerged. The tolerance for this dimension has been taken as ± 0.02 cm, the same as given in Table 1 for the Engler viscosimeter.

Meissner's formula for calculating the average head may be written

$$\text{Average head, } h = \frac{h_1 - h_2}{\log_e\left(\frac{h_1}{h_2}\right)} \qquad (1)$$

where h_1 and h_2 are the initial and final heads, respectively. This formula is unsatisfactory when the velocity of flow is high,[8] but it is the best available and was used to calculate the average head in Tables 2 and 3.

The diameter of the container, D, is of importance because it enters indirectly into equation (1), being necessary to obtain h_2 by the formula

$$\frac{\pi}{4} D^2 (h_1 - h_2) = 60, \qquad (2)$$

60 cm³ being the volume discharged in time, t. The so-called Saybolt viscosity is the time, t, expressed in seconds. It follows from equation (2) that when D has the minimum value of 2.955 cm, $h_1 - h_2$ is equal to 8.75 cm. Therefore the cylindrical part of the container must have at least this height. Meissner found it to be 10.35 cm, while it is about 9.7 cm in the standard Saybolt Universal viscosimeter.

[8] T. P. 100, p. 40.

6. EQUATION FOR DETERMINING VISCOSITY FROM DIMENSIONS OF EFFLUX INSTRUMENT AND THE TIME OF DISCHARGE

Efflux instruments, such as the Saybolt Universal and the Engler viscosimeters, may be used to determine kinematic viscosity, which is the name given to the ratio of absolute viscosity to the density. If absolute viscosity is required, it can be obtained by multiplying the kinematic viscosity by the density, which must be determined by an auxiliary instrument.

Kinematic viscosity may be calculated by the equation [9]

$$\frac{\mu}{\gamma} = \frac{\pi\, g d^4 h}{128\, Q\, (l + \lambda)} \left(t - \frac{m v^2}{g} \right) \tag{3}$$

where μ is in poises, the cgs unit of viscosity, γ is the density in grams per cubic centimeter, d and l are the diameter and length, respectively, of the outlet tube, in centimeters; Q is the volume in cubic centimeters, discharged in the time, t, in seconds; λ is the "Couette correction," which must be added to the measured length of tube to get the effective length; h is the average head, in centimeters, of liquid of density, γ; and m is the coefficient of the kinetic energy correction, $\frac{m v^2}{g}$, g being the acceleration due to gravity, or 981 cm per second, per second, and v the mean velocity in centimeters per second.

Table 3 gives all values needed for finding the kinematic viscosity from equation (3) with the exception of λ and m. Unfortunately, the value of 1.12 for m, which is commonly used with long-tube viscosimeters, [10] can not be used for the standard Saybolt Universal viscosimeter where the ratio of length to diameter of the outlet tube is only 6.94. The Couette correction is also unknown, because it can have neither the value of zero, which is generally employed with long-tube viscosimeters, nor can it be equal to 0.82 d, which, according to Higgins,[11] is its value in the case of submerged discharge.

If equation (3) is written in the form

$$\frac{\mu}{\gamma} = A\, t - \frac{B}{t}, \tag{4}$$

it is evident that A and B are instrumental constants which may be obtained by finding the time of discharge for two liquids

[9] T. P. 100, p. 6.
[10] E. C. Bingham and R. F. Jackson, Scientific Paper No. 298, Bureau of Standards, p. 65; 1917.
[11] W. F. Higgins, Collected Researches, Nat. Phys. Lab., 11, p. 9; 1914.

having, preferably, a considerable difference in viscosity. Values of the unknown quantities in equation (3) may then be calculated by combining equations (3) and (4). It was found in this way that for Saybolt instruments Nos. 580 and 727, the Couette correction had a value of 0.4 d, while m was equal to 0.97.[12] These instruments were not of standard dimensions and it is not known upon what the values of λ and m depend, but presumably these constants would be about the same for a standard Saybolt Universal viscosimeter, as for instruments Nos. 580 and 727.

7. HIGGINS'S METHOD FOR DETERMINING THE RELATION BETWEEN KINEMATIC VISCOSITY AND TIME OF DISCHARGE

As indicated by Higgins, if both sides of equation (4) are divided by t, and values of $\frac{\mu}{\gamma t}$ as ordinates are plotted against values of $\frac{1}{t^2}$, a straight line will be obtained if A and B are constants. The value of A may be read on the scale of ordinates at the point where the calibration curve intersects the axis of ordinates, and the tangent of the acute angle between the extended straight portion of the calibration curve and the axis of abscissas gives the value of B.

Table 4 shows times of discharge obtained with the four Saybolt instruments, each time, both in this table and in Table 5, being the average for 10 runs. With the exception of olive oil, which has the highest viscosity of any liquid listed in Table 4, and the 60 per cent sugar solutions which also had a high viscosity, the difference between the maximum and the minimum time, for any of the four instruments, exceeded 2 per cent only twice out of 24 tests, both cases being for water at 20° C (68° F).

Fig. 1 has been plotted from data of Tables 4 and 5. The bend in the calibration curve indicates that the flow is turbulent when water is used, so that A and B have different values than for viscous flow. Therefore water is not a suitable calibrating liquid for these viscosimeters. Fig. 1 also shows a disagreement between tests made with solutions of ethyl alcohol, sucrose, and glycerol. The kinematic viscosities of glycerol solutions were calculated from data of Archbutt and Deeley,[13] while the kinematic viscosities of distilled water, ethyl alcohol solutions, and sucrose solutions

[12] T. P. 100, p. 30; Proc., A. S. T. M., 17 (II), p. 562; 1917.

[13] L. Archbutt, and R. M. Deeley, Lubrication and Lubricants, p. 161; 1912.

were taken from data of Bingham and Jackson.[14] As pointed out by Upton [15] there is a doubt as to the accuracy of Archbutt and Deeley's values for glycerol solutions. Other reasons for discarding the tests with these solutions are considered in the Appendix.

TABLE 4.—Comparison of Four Standard Saybolt Viscosimeters

LIQUIDS OF KNOWN VISCOSITY

Liquid	Temperature	Time of discharge for instrument No.—			
		108	109	110	111
	°C	Sec.	Sec.	Sec.	Sec.
Water	15	30.82	31.34	31.42	31.22
Do	20	30.90	30.78	30.10	30.88
Do	20	30.72	31.02	30.12	30.28
Do	50	28.76	28.76	28.56	28.68
Do	75	28.22	28.06	28.04	28.06
10 per cent ethyl alcohol	15	33.50	33.16	33.00	33.48
Do	20	32.06	32.12	31.98	31.96
30 per cent ethyl alcohol	20	35.24	35.24	35.10	35.24
Do	20	35.68	35.64	35.82	35.74
50 per cent ethyl alcohol	Zero	.....	.....	.....	53.34
Do	15	38.88	38.90	39.12	38.98
Do	15	39.40	39.50	39.08	38.92
Do	20	36.74	36.76	36.52	36.54
20 per cent sucrose, old	20	34.16	34.12	33.88	34.06
20 per cent sucrose, fresh	20	33.62	33.68	33.70	33.58
40 per cent sucrose, old	20	45.26	45.56	44.78	45.08
40 per cent sucrose, fresh	20	44.66	45.10	44.52	44.64
60 per cent sucrose, old	20	186.68	190.52	193.66	194.38
60 per cent sucrose, fresh	20	217.50	213.30	212.92	214.18

LIQUIDS OF UNKNOWN VISCOSITY

Olive oil	± 22	478.80	481.80	475.26	461.30
Three-fourths spindle oil	.....	58.10	58.30	58.04	58.22
One-fourth kerosene	.....				
Press oil No. 3	15	54.66	53.86	53.62	54.16
Do	20	50.28	49.86	49.76	49.80
Do	25	48.54	48.18	48.38	48.24
Meter oil	20	65.78	65.50	65.58	65.52
Press oil No. 2	25	97.14	97.46	95.38	96.04
Press oil No. 4	25	115.32	113.98	114.74	114.98
Press oil No. 1	25	171.38	171.58	171.88	170.80

[14] Kinematic viscosities of these liquids, calculated from data of Bingham and Jackson and other experimenters, are given in the Appendix of T. P. 100, p. 52.

[15] G. E. Upton, Proc., A. S. T. M., **15** (1), p. 323; 1915.

TABLE 5.—Comparison of Two Standard Saybolt Universal Viscosimeters with Engler Instrument No. 2204 U

Liquid	Temperature (approximate)	Time of discharge			Kinematic viscosity from Engler instrument
		Saybolt No.—		Engler[a]	
		110	111		
	°C	Sec.	Sec.	Sec.	Poises g/cm³
Olive oil	23	463.6	483.8	712.0	1.042
Cer oil No. 8	23	313.9	317.6	471.1	.684
Transformer oil	21	122.7	123.3	185.9	.253
Floor oil	22	178.7	182.0	270.3	.383
Paraffin oil	20	684.4	692.2	1006.7	1.476
Three-fourths kerosene, one-fourth spindle	19	38.4	38.9	63.7	.0349
One-half kerosene, one-half spindle	20	45.3	45.3	74.2	.0587
One-fourth kerosene, three-fourths spindle	19	61.6	61.6	98.1	.1067
Spindle oil	21	88.8	90.8	139.0	.1774
Press oil No. 3	21	49.5	50.0	81.3	.0735
Meter oil	22	63.3	63.4	101.1	.1115
Old sucrose solution	17	253.9	254.2	399.9	.579
Fresh sucrose solution, 60 per cent	16	285.0	286.7	451.6	.656
Fresh sucrose solution, 40 per cent	17	46.9	46.6	76.8	.0642
Press oil No. 2	23	106.3	105.7	160.5	.2127
Press oil No. 4	22	137.3	135.6	205.6	.2838
Press oil No. 1	24	187.9	187.7	283.0	.403
Oil No. 885	23	183.3	185.2	274.8	.390
Oil X_3	24	278.8	274.9	413.1	.598
Spindle oil	25	83.5	84.0	129.9	.1622
Oil No. 458	26	251.5	250.3	380.7	.550
Oil X_2	27	336.1	336.2	501.8	.731
Glycerol solution, 52.2 per cent	20	.....	46.8	70.4	.0633
Glycerol solution, 70.0 per cent	20	.....	105.6	153.8	.2018
Glycerol solution, 80.3 per cent	20	212.9	211.9	328.0	.471
Glycerol solution, 85.0 per cent	20	.....	428.3	658.3	.962
Glycerol solution, 90.1 per cent	20	806.5	792.7	1231.1	1.806
Glycerol solution, 96.0 per cent	20	.....	1965.8	2950.6	4.336

[a] The times, Engler, for the glycerol solutions, were obtained by using an abnormal filling and a conversion factor, and are probably less reliable than the other readings.

The tests with sucrose solutions, while consistent enough with one another, would indicate a lower position of the calibration curve than obtained by tests with alcohol solutions. It is believed that this difference is due to the difference in surface tension, and that a calibrating liquid should be selected with a surface tension most nearly equal to that of oils, since Saybolt viscosimeters are mainly used for testing oils. Table 6 shows that 30 or 50 per cent ethyl alcohol solutions have a suitable surface tension.[16] On the other hand, it may be seen from Table 7 that the surface tension of sucrose solutions is much too high.[17]

[16] From Landolt-Bornstein, Physikalisch chemische Tabellen, 4th ed., pp. 124, 128.

[17] Calculated from data of E. O. von Lippmann, Die Chemie der Zuckerarten, 2, p. 1119; 1904.

TABLE 6.—Surface Tension of Oils and Ethyl Alcohol Solutions, from Landolt-Börnstein

Liquid	Temperature at which density was taken	Density	Temperature at which surface tension was taken	Surface tension, in dynes per centimeter
	°C		°C	
Olive oil	20	0.910	20	31.74
		.917	20	34.5
		.9136		32.09
		.9136	25.8	36.9
		.9136	54	30.56
Do	15	.989	15	35.6
			20	35.4
			25	34.9
			30	34.4
Russian illuminating oil		.822	18	30.5
				32.3
American mineral oil		.756	18	29.4
				30.4
Ethyl alcohol, 0 per cent			15	72.2
Ethyl alcohol, 10 per cent			15	51.2
Ethyl alcohol, 20 per cent			15	40.6
Ethyl alcohol, 30 per cent			15	34.7
Ethyl alcohol, 40 per cent			15	31.2
Ethyl alcohol, 50 per cent			15	29.1
Ethyl alcohol, 60 per cent			15	27.7

TABLE 7.—Surface Tension of Sucrose Solutions at 18.5° C (65.3° F) According to E. O. von Lippmann

Per cent sucrose	Surface tension in dynes per centimeter	Per cent sucrose	Surface tension in dynes per centimeter	Per cent sucrose	Surface tension in dynes per centimeter	Per cent sucrose	Surface tension in dynes per centimeter
0	72.8	20	67.8	40	63.4	60	59.4
5	71.5	25	66.7	45	62.4	65	58.4
10	70.3	30	65.7	50	61.4	70	57.5
15	69.1	35	64.5	55	60.4		

Since, for various reasons which have been given, distilled water, glycerol, and sucrose solutions are not suitable calibrating liquids for short-tube viscosimeters having a discharge into the air, tests with alcohol solutions must be given the most weight. Yet alcohol solutions, alone, are not sufficient to determine the values of A and B in equation (4), as the viscosities are too low; and if Higgins's method were used, an excessive extrapolation would be required. It is therefore necessary to find the constant A by other means, using Higgins's method for finding B, and for a rough check on the value of A.

8. A METHOD OF FINDING THE CONSTANT *A* IN EQUATION (4)

In order to obtain points on Fig. 1, it is necessary either to use liquids for which viscosities and densities are known for the temperatures employed, or to determine the kinematic viscosity by means of an instrument for which the constants *A* and *B* are known. After it had been found that the most viscous liquid which was available for calibrating purposes was a 50 per cent ethyl alcohol solution, and that no systematic difference in readings

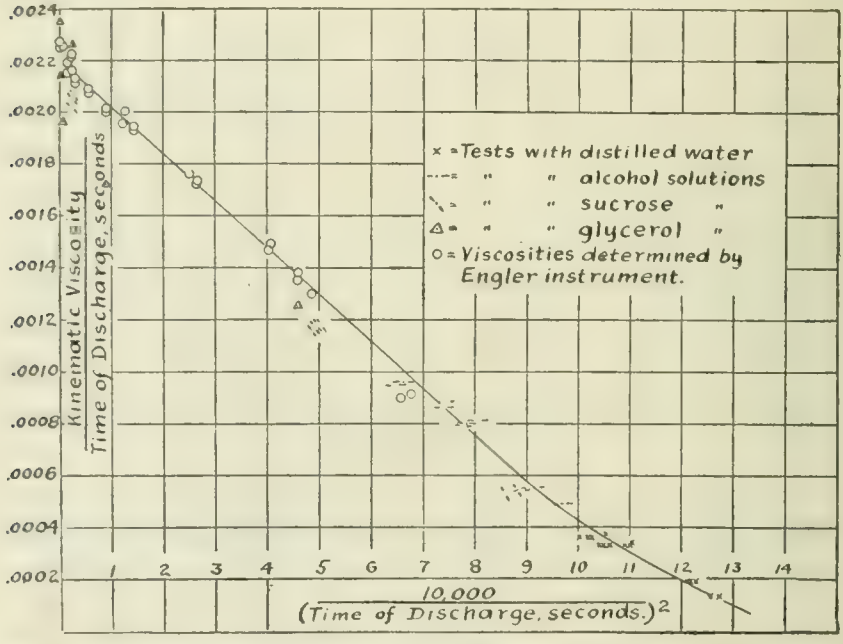


FIG. 1.—*Calibration curve of standard Saybolt Universal viscosimeter, by Higgins's method*

of the four Saybolt instruments could be detected, it was decided to use the method of viscosimeter comparison without temperature control [18] taking Saybolt instruments Nos. 110 and 111 and Engler No. 2204 U. Table 5 shows the results of these tests, and gives the kinematic viscosities calculated from the Engler time of discharge, using the equation.[19]

$$\frac{\mu}{\gamma} = 0.00147\, t - \frac{3.74}{t} \qquad (5)$$

[18] T. P. 100, p. 19.

[19] T. P. 100, p. 31. (This equation was derived from Fig. 4, and was not based on the tests with sucrose solutions shown in Fig. 2, which were few and discordant.)

If the kinetic energy correction (i. e., the last term) is negligible, and t_e and t_s are the times of discharge for the Engler and Saybolt viscosimeters, respectively, then

$$\frac{t_e}{t_s} = \frac{A}{0.00147} \tag{6}$$

A being the desired constant for the Saybolt instrument. Values of $\frac{t_e}{t_s}$, as calculated from data of Table 5, are shown in Fig. 2. The points for alcohol solutions are calculated from data in Table 4 taken in connection with Engler times of flow previously obtained.[20] As the kinetic energy correction decreases, the time ratio decreases

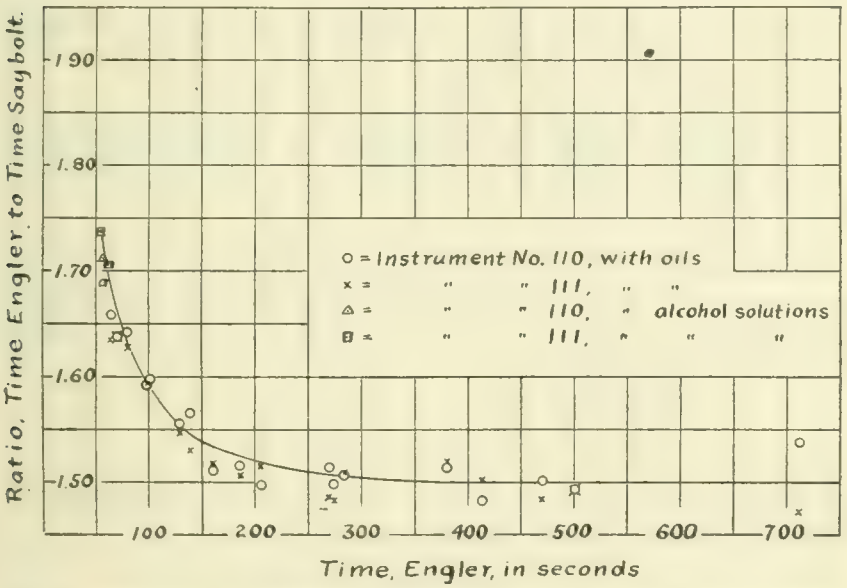


FIG. 2.—*Ratio of times of discharge of Engler viscosimeter No. 2204 U and the standard Saybolt Universal viscosimeter*

until, when the kinetic energy correction is negligible, the ratio becomes a constant. Thus the lowest ratio obtained should be the constant value required. The most viscous oil used gave a time ratio of 1.472 and 1.455, respectively, for instruments Nos. 110 and 111. These values are probably too low on account of experimental error. On the other hand, the average of 12 oils of high viscosity gives 1.503 and 1.495, which would be slightly too high if the kinetic energy correction was not negligible in all cases. If the average value 1.499 is used, then from equation (6), $A = 0.00220$.

[20] T. P. 100, p. 24.

9. DETERMINATION OF THE CONSTANT *B* BY HIGGINS'S METHOD

The value of *A*, as determined above, fixes one end of the calibration curve of Fig. 1, which must pass through the point with an ordinate of 0.00220 and an abscissa of zero. The rest of the curve has been located by the tests with distilled water and alcohol solutions of Table 4 and the tests of Table 5. It is seen that the line obtained is nearly parallel to the one indicated by the tests with sucrose solutions, and that the tangent of the acute angle formed by this line with the axis of abscissas, which determined the value of *B*, is about 1.8.

This value of *B*, and that of *A* given above, were found without reference to the dimensions of the instruments. A check of these values by Herschel's method will serve also as a check on the accuracy with which the dimensions were measured.

10. DETERMINATION OF THE COUETTE CORRECTION AND THE KINETIC ENERGY CORRECTION BY HERSCHEL'S METHOD

By substituting the dimensions given in Table 2 in equation (3) equations may be found for the different Saybolt instruments. This was done for instruments Nos. 108 to 111, and the average for the four instruments was found to be

$$\frac{\mu}{\gamma}(1.229 + \lambda) = 0.00280t - \frac{2.387}{t}m. \tag{7}$$

If the Couette correction and the kinetic energy correction are both disregarded in equation (3),

$$\mu' = \frac{\pi}{128}\frac{gd^4}{Q}\frac{\gamma}{l}\frac{h}{}t. \tag{8}$$

Then if "Reynolds's criterion," $\frac{v\,d\,\gamma}{\mu}$, is plotted against $\frac{\mu'}{\mu}$, it may be shown that [21]

$$\lambda = \left(\frac{\mu'}{\mu} - 1\right)l. \tag{9}$$

$$m = \frac{32\,l}{\tan\theta\,d}. \tag{10}$$

The value of $\frac{\mu'}{\mu}$ corresponding to a value of Reynolds's criterion of zero must be used in equation (9); that is, $\frac{\mu'}{\mu}$ must be found

[21] T. P. 100, p. 13

by prolonging the calibration curve until it intersects the axis of abscissas. In equation (10) θ is the angle between the calibration curve and the axis of abscissas.

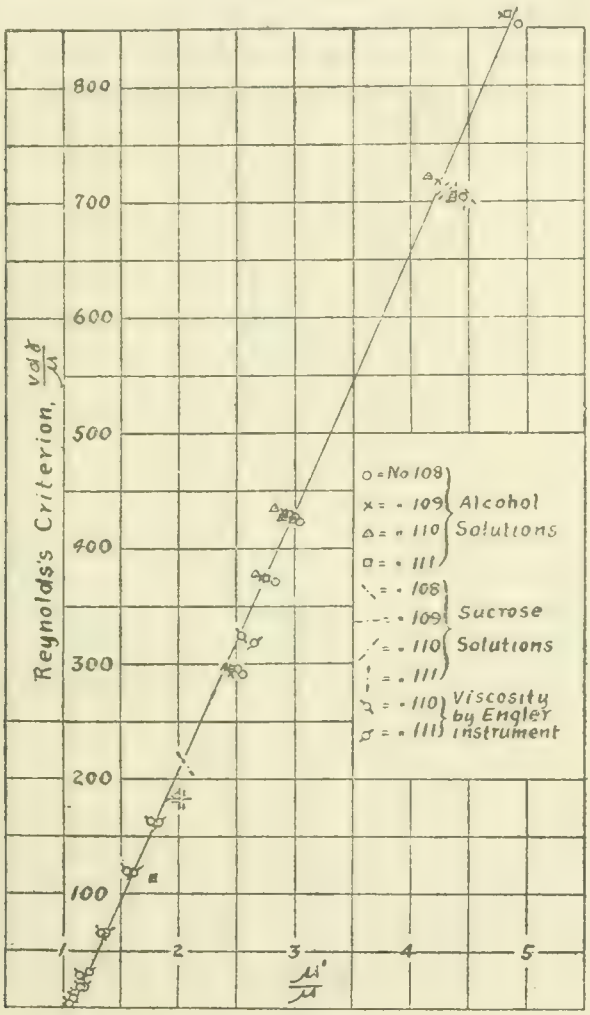


FIG. 3.—*Calibration curve of Standard Saybolt Universal viscosimeter, by Herschel's method*

Fig. 3 was calculated from the data of Tables 4 and 5, while Fig. 4 gives an enlarged view of the lower end of the calibration curve for use in finding the Couette correction. By extrapolation, $\frac{\mu'}{\mu}$ has a value of 1.044 for instrument No. 110, and 1.090 for instrument No. 111, giving an average of 1.067. These values,

if obtained approximately, merely by averaging the nine lowest values obtained by experiment, would have been 1.058, 1.104, and 1.081, respectively. These latter results are, of course, too high, as Reynolds's criterion must be greater than zero in all tests.

From equation (9) the Couette correction is 0.067 (1.229) =

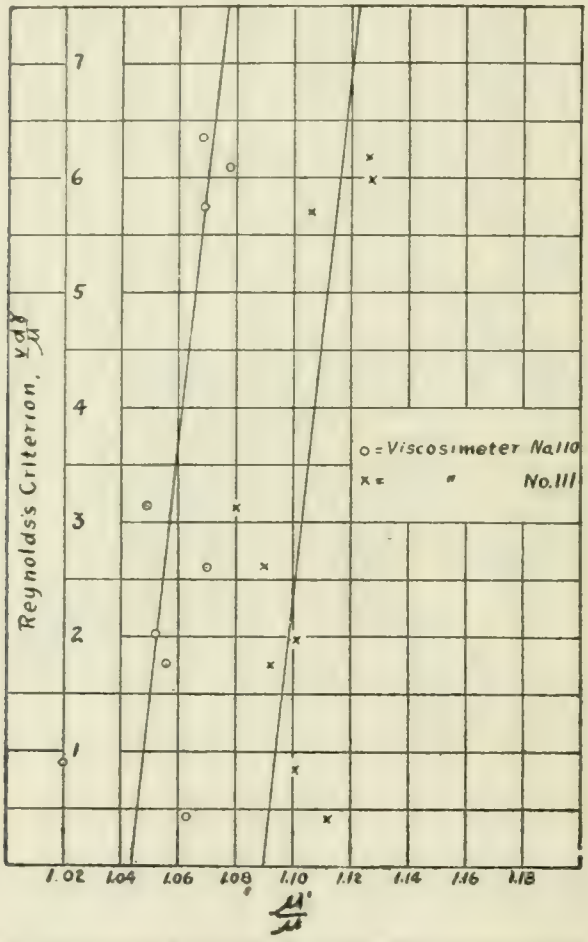


FIG. 4.—*Enlarged view of bottom of Fig. 3, used to determine the Couette correction*

0.0824, or, since it is usually expressed as proportional to the diameter, $\lambda = 0.466\ d$. When the kinetic energy correction is negligible, combining equations (4) and (7) gives

$$\frac{\mu}{\gamma} = A\ t = \frac{0.00289}{(1.229+\lambda)} t \qquad (11)$$

from which, with λ equal to 0.082, the value of A is found to be 0.00220.

From Fig. 3 tan θ is 225, so that by equation (10) $m = 0.989$. By combining the last terms of equations (4) and (7)

$$\text{kinetic energy correction} = \frac{B}{t} = \frac{2.387\, m}{(l+\lambda)\, t} \qquad (12)$$

from which, with $m = 0.989$ and $\lambda = 0.082$, the value of B becomes 1.801. The complete equation for determining the kinematic viscosity from the time of discharge is therefore

$$\frac{\mu}{\gamma} = 0.00220\, t - \frac{1.80}{t} \qquad (13)$$

which applies to a Saybolt Universal viscosimeter of normal dimensions.

11. EQUATIONS FOR INSTRUMENTS NOT OF NORMAL DIMENSIONS

According to Ubbelohde, the kinematic viscosity may be determined from the time of discharge of the Engler viscosimeter by the equation

$$\frac{\mu}{\gamma} = \left(4.072\frac{t}{t_w} - 3.513\frac{t_w}{t}\right) 0.01797 \qquad (14)$$

where t_w is the time of discharge for 200 cm³ of water at 20° C. (68° F.), commonly called the water rate. Comparing equations (4) and (13) it is seen that Ubbelohde assumes that A is inversely and B is directly proportional to the water rate. Now, in reality [22]

$$t_w = \frac{\frac{\mu}{\gamma}}{2A}\left(1 + \sqrt{1 + \frac{4AB}{\left(\frac{\mu}{\gamma}\right)^2}}\right) \qquad (15)$$

so that there is little justification for Ubbelohde's assumptions. It therefore seems preferable to fall back on equation (3), and to assume that the slight variations in dimensions allowed in Table 3 will not cause any change in the values of m and of $\frac{\lambda}{d}$. This assumption has some justification in the slight difference between the values of these quantities, as found with instruments Nos. 108 to 111 and with Nos. 580 and 727. Taking, accordingly, the values of $\frac{\lambda}{d}$ and m, as found above, and the limiting viscosimeter dimensions from Table 3, limiting equations may be found for instruments of standard, though not of the average or normal dimensions. Equation (16) was calculated by selecting the

[22] Proc., A. S. T. M., 15 (1), p. 318; 1915.

dimensions which would tend to give a minimum time of discharge while equation (17) was calculated from dimensions to give a maximum time.

$$\frac{\mu}{\gamma} = 0.00234\,t - \frac{1.82}{t} \text{ (minimum)} \tag{16}$$

$$\frac{\mu}{\gamma} = 0.00204\,t - \frac{1.79}{t} \text{ (maximum)} \tag{17}$$

12. APPLICATIONS OF THE NEW EQUATIONS

Table 8 has been calculated from equations (13), (16), and (17).

TABLE 8.—Times of Discharge for Standard Saybolt Universal Viscosimeters

Liquid	Temperature	Kinematic viscosity	Time of discharge		
			Minimum	Normal	Maximum
	°C		Sec.	Sec.	Sec.
30 per cent ethyl alcohol	20	0.02841	34.6	35.7	37.4
Do	25	.02292	33.2	34.2	35.7
50 per cent ethyl alcohol	20	.03144	35.4	36.6	38.4
Do	25	.02636	34.2	35.2	36.8
A heavy oil		1.00	428.0	455.0	490.0

It should be noted that the table is based on the assumption that the departure of each dimension from the normal was of such sign as to produce the maximum possible deviation from the normal time of flow. In practice, however, the departures would tend to counteract one another, so that the variations in times of discharge would not be as great as shown in Table 8. The heavy oil has been assumed of a high enough viscosity to make the kinetic energy correction negligible. Water is not recommended for a calibrating liquid, and the time of discharge for water has accordingly been omitted.

Equations for calculating kinematic viscosity serve as a ready means for calculating conversion tables between times of discharge of different types of viscosimeters. Thus, equations (5) and (13) have been used to calculate Table 9 for conversions between the Saybolt Universal viscosimeter of normal dimensions and Engler viscosimeter No. 2204 U. All previously published conversion tables or diagrams for Saybolt Universal viscosimeters are based on tests with instruments of obsolete dimensions and should therefore be discarded.[23]

[23] T. Peckham, p. ; T. B. Stillman, Examination of Lubricating Oils, p. 22, 1914; R. F. Bacon and W. A. Hamor, The American Petroleum Industry, I, p. 111, 1916.

TABLE 9.—Conversion Table for Viscosimeters

[Engler No. 1221 U and standard Saybolt Universal]

Time, Engler	Time, Saybolt	Time, ratio	Engler degrees = time, Engler 51.3[a]	Kinematic viscosity	Time, Engler	Time, Saybolt	Time, ratio	Engler degrees = time, Engler 51.7	Kinematic viscosity
Sec.	Sec.				Sec.	Sec.			Poises / g/cm³
56	32.4	1.73	1.09	0.0155	130	83.6	1.56	2.54	0.1624
58	33.7	1.72	1.13	.0208	140	90.6	1.55	2.73	.1793
60	35.1	1.71	1.17	.0259	150	97.4	1.54	2.93	.1956
62	36.5	1.70	1.21	.0308	160	104.4	1.53	3.12	.2121
64	37.8	1.69	1.25	.0356	180	117.7	1.53	3.51	.2437
66	39.2	1.68	1.29	.0403	200	131.5	1.52	3.90	.2753
68	40.6	1.68	1.33	.0448	225	148.3	1.52	4.39	.3140
70	42.0	1.67	1.36	.0495	250	165.3	1.51	4.88	.3523
75	45.5	1.65	1.46	.0603	275	182.0	1.51	5.36	.3904
80	49.0	1.63	1.56	.0709	300	199.0	1.51	5.85	.4282
85	52.4	1.62	1.66	.0810	325	215.6	1.51	6.34	.4660
90	55.8	1.61	1.76	.0905	350	232.9	1.50	6.82	.5038
95	59.4	1.60	1.85	.1003	375	249.2	1.50	7.31	.5413
100	62.8	1.59	1.95	.1095	400	266.1	1.50	7.80	.5784
110	69.8	1.58	2.15	.1278	500	333.0	1.50	9.75	.7271
120	76.8	1.56	2.34	.1453	600	400.0	[b] 1.50	11.70	.8753

[a] See T. P. 100, p. 25.

[b] This value holds good for all higher viscosities.

13. THE REDWOOD VISCOSIMETER

It is sometimes necessary for the export trade to consider the Redwood viscosimeter. This instrument has never been standardized with respect to its dimensions, and the standardization with rapeseed oil is unsatisfactory,[24] as it is equivalent to fixing the value of A in equation (4) and leaving B undetermined. Therefore all equations for the Redwood instrument [25] must be regarded as approximations, and it is probable that these instruments would not give as concordant results with liquids of low viscosity as with the rapeseed oil with which a standard time of discharge is obtained.

If Higgins's equation is taken as probably as good as any, and equation (5) is used for the Engler viscosimeter, Table 10 may be obtained to supplement Table 9.

[24] L. Archbutt and R. M. Deeley, Lubrication and Lubricants, p. 108, 1912.

[25] T. P. 100, p. 31.

TABLE 10.—Conversion Table for Redwood, Saybolt, and Engler Viscosimeters

Time, Engler	Time, ratio			Time, Redwood	Time, Engler	Time, ratio			Time, Redwood
	Engler-Saybolt	Engler-Redwood	Saybolt-Redwood[a]			Engler-Saybolt	Engler-Redwood	Saybolt-Redwood[a]	
Sec.				Sec.	Sec.				Sec.
56	1.73	2.61	1.51	21.5	140	1.55	1.81	1.17	77.5
58	1.72	1.93	1.12	30.0	150	1.54	1.80	1.17	83.2
60	1.71	1.93	1.13	31.2	160	1.53	1.80	1.17	89.1
62	1.70	1.92	1.13	32.2	180	1.53	1.80	1.17	100.3
64	1.69	1.91	1.13	33.4	200	1.52	1.79	1.18	111.9
66	1.68	1.91	1.13	34.6	225	1.52	1.79	1.18	126.0
68	1.68	1.90	1.14	35.8	250	1.51	1.78	1.18	140.3
70	1.67	1.90	1.14	36.9	275	1.51	1.78	1.18	154.4
75	1.65	1.88	1.14	39.8	300	1.51	1.78	1.18	168.5
80	1.63	1.87	1.15	42.7	325	1.51	1.78	1.18	183.0
85	1.62	1.86	1.15	45.7	350	1.50	1.78	1.18	197.0
90	1.61	1.86	1.15	48.5	375	1.50	1.77	1.18	211.3
95	1.60	1.85	1.16	51.4	400	1.50	1.77	1.18	225.5
100	1.59	1.84	1.16	54.3	500	1.50	1.77	1.18	282.0
110	1.58	1.83	1.16	60.1	600	1.50	1.77	1.18	339.0
120	1.56	1.82	1.16	66.0					
130	1.56	1.81	1.17	71.7					

[a] These values were calculated directly from the times of discharge and differ in some cases by 0.01 from values calculated from the two previous columns.

14. CONCLUSION

Table 3 and equations (13), (16), and (17) give the complete standardization of the Saybolt Universal viscosimeter. It has previously been impossible to determine whether a Saybolt Universal viscosimeter gave normal readings, as neither the dimensions nor normal times of flow for any given liquids were known. Now that these data have been determined, limit gages have been prepared, and the Bureau of Standards is now in a position to certify whether or not a given instrument is of standard dimensions.

WASHINGTON, February 3, 1919.

APPENDIX

THE VISCOSITY OF GLYCEROL SOLUTIONS

Archbutt and Deeley give viscosities of glycerol solutions of various specific gravities. Different experimenters do not agree very closely in regard to the relation between the density and the concentration of glycerol solutions. Table 11 is based on data of Gerlach [26] and of Archbutt and Deeley.

TABLE I.—Densities and Viscosities of Glycerol Solutions at 20° C (68° F) According to Archbutt and Deeley and Gerlach

Per cent glycerol	Density in grams per cubic centimeter	Viscosity in poises	Kinematic viscosity	Per cent glycerol	Density in grams per cubic centimeter	Viscosity in poises	Kinematic viscosity
5	1.0098	0.01181	0.01170	55	1.1393	0.07664	0.06727
10	1.0217	.01364	.01335	60	1.1528	.1031	.08943
15	1.0337	.01580	.01529	65	1.1662	.1451	.1244
20	1.0461	.01846	.01765	70	1.1797	.2149	.1822
25	1.0590	.02176	.02055	75	1.1932	.3371	.2825
30	1.0720	.02585	.02411	80	1.2066	.5534	.4586
35	1.0855	.03115	.02870	85	1.2201	1.025	.8401
40	1.0989	.03791	.03450	90	1.2335	2.076	1.683
45	1.1124	.04692	.04218	95	1.2465	4.801	3.852
50	1.1258	.05908	.05248				

To avoid the effect of possible error in Gerlach's relation between density and concentration, solutions were made up to approximately the desired concentration by the above table, and then the viscosities of the solutions were interpolated from the table after the exact density had been found.

It was noted that the 80 per cent solution evaporated, while the 90 per cent solution absorbed moisture. Consequently, if it were not for the doubt as to the accuracy of Archbutt and Deeley's values of the viscosity, which appear to be especially erratic for concentrations in this neighborhood, solutions of about 85 per cent would be the most valuable for calibrating purposes, because the viscosity would remain most constant when exposed to the air in technical viscosimeters.

A much greater range of viscosities may be obtained with glycerol than with alcohol or sucrose solutions. Comparing kinematic viscosities at 20° C (68° F), the maximum which can be obtained with ethyl alcohol is 0.03144 with a 50 per cent solution, while practically the maximum obtainable with sucrose is 0.4392 for a 60 per cent solution. That a 60 per cent solution has approximately the greatest concentration which can be employed is shown by a table given by von Lippmann [27] for the per cent of sucrose in saturated solutions at various temperatures. This shows an increase from 64.18 per cent at 0° C (32° F) to 82.97 per cent at 100° C (212° F), the value at 20° C (68° F) being 67.09 per cent.

2. THE VISCOSITY OF CASTOR OIL

On account of the uncertainty in regard to the viscosity of glycerol solutions and the rapid change of viscosity with the concentration, it is probable that the best calibrating liquid for very high viscosities is castor oil, although this can not be regarded as

[26] G. T. Gerlach, Chemische Industrie 7, p. 277; 1884.

[27] E. O. von Lippmann, Chemie der Zuckerarten, 2, p. 1089; 1904.

entirely satisfactory on account of slight differences between different samples. However, the difficulty of accurate determinations of the viscosity of such a very viscous liquid is so great that a viscosity determination may well be considered as a test of the skill of the experimenter rather than as a test of the variation between samples. The extent of this variation may be judged from the work of Deering and Redwood [28] on 23 samples of Indian oil. Their results were rearranged in the order of the density, but no regular variation of viscosity with the density could be observed. Table II shows the maximum and minimum values for the 23 samples.

TABLE II.—Density and Viscosity of Castor Oil According to Deering and Redwood

Specific gravity at 60° F (15.6° C)	Density at 60° F	Time of discharge, in seconds, Redwood viscosimeter, at 100° F (37.8° C)	Kinematic viscosity at 100° F
0.9637 minimum	0.9628	1190 maximum	3.093
0.9639	.9630	1160 minimum	3.015
0.9642 maximum	.9638	1174	3.051

The kinematic viscosities were determined by Higgins's equation.[29]

$$\frac{\mu}{\gamma} = 0.00260\ t - \frac{1.715}{t}. \qquad (18)$$

A slight error might be caused by deterioration of the oil with time. Lewkowitsch [30] says:

"Properly refined castor oil keeps very well and does not easily turn rancid, as observations made in the author's laboratory have shown. A sample exposed to the atmosphere for four years contained only 1 per cent of free fatty acids."

Another or possibly the same sample, which was exposed for four years, had its specific gravity at 60° F (15.6° C) increased from 0.9591 to 0.9629.

Probably the most accurate determinations of the viscosity of castor oil have been made by Kahlbaum and Räber.[31] Their results are given in Table III, to which the kinematic viscosities have been added.

TABLE III.—Density and Viscosity of Castor Oil According to Kahlbaum and Räber

Temperature in degrees centigrade	Density in grams per cubic centimeter	Viscosity in poises	Kinematic viscosity	Temperature in degrees centigrade	Density in grams per cubic centimeter	Viscosity in poises	Kinematic viscosity
5	0.9707	37.60	38.74	23	.9583	7.67	8.00
6	.9700	34.475	35.54	24	.9576	7.06	7.37
7	.9693	31.56	32.56	25	.9569	6.51	6.80
8	.9686	28.90	29.84	26	.9562	6.04	6.32
9	.9679	26.45	27 33	27	.9555	5.61	5.87
10	.9672	24.18	25.00	28	.9548	5.21	5.46
11	.9665	22.075	22.84	29	.9541	4.85	5.08
12	.9659	20.075	20.78	30	.9534	4.51	4.73
13	.9652	18.25	18.91	31	.9527	4.21	4.42
14	.9645	16.61	17.22	32	.9520	3.94	4.14
15	.9638	15.14	15.71	33	.9513	3.65	3.84
16	.9631	13.805	14.33	34	.9506	3.40	3.58
17	.9624	12.65	13.14	35	.9499	3.16	3.33
18	.9617	11.625	12.09	36	.9492	2.94	3.10
19	.9610	10.71	11.15	37	.9485	2.74	2.89
20	.9603	9.86	10.27	38	.9478	2.58	2.72
21	.9596	9.06	9.44	39	.9471	2.44	2.58
22	.9589	8.34	8.70	40	.9464	2.31	2.44

[28] W. H. Deering and Boverton Redwood, Jour. Soc. for Chem. Industriy **18**, p. 959; 1894.

[29] W. F. Higgins, Collected Researches, Nat. Phy. Lab., **11**, p. 12, 1914.

[30] J. Lewkowitsch, Chemical Technology and Analysis of Oils, Fats, and Waxes, **2**, pp. 395–396; 1915.

[31] W. A. Kahlbaum and S. Räber, Abd. der Kaiserl. Leop.-Carol. Deutschen Akademie der Naturforscher **84**, p. 201; 1905.

Castor oil has the advantage, as compared with glycerol and sucrose solutions, of evaporating much less easily, and hence it may be used over a much greater range of temperatures. Table IV gives viscosities as determined by Archbutt and Deeley,[32] together with the kinematic viscosities which have been calculated from their data.

TABLE IV.—Density and Viscosity of Castor Oil According to Archbutt and Deeley

Temperature		Viscosity in poises	Density in grams per cubic centimeter	Kinematic viscosity
Degrees Fahrenheit	Degrees centigrade			
100	37.8	2.729	0.9473	2.881
150	65.6	.605	.9284	.6517
212	100.0	.169	.9050	.1867

By interpolation in Table II the density according to Kahlbaum and Räber would be 0.9634 at 15.6° C, which may be compared with Redwood and Deering's values in Table I, while the density at 37.8° C would be 0.9479, which agrees fairly well with Archbutt and Deeley's value. Similarly, Kahlbaum and Räber give a kinematic viscosity of 2.75 at 37.8° C, which may be compared with the values obtained by the other experimenters. It would appear that while the variation in density and viscosity is appreciable, it is too small to be of importance with short-tube technical instruments.

[32] L. Archbutt and R. M. Deeley, Lubrication and Lubricants, pp. 186–188; 1912.

DEPARTMENT OF COMMERCE

TECHNOLOGIC PAPERS

OF THE

BUREAU OF STANDARDS

S. W. STRATTON, DIRECTOR

No. 113

DETERMINATION OF PERMEABILITY OF BALLOON FABRICS

BY

JUNIUS DAVID EDWARDS, Associate Chemist

Bureau of Standards

ISSUED JULY 2, 1918

PRICE, 10 CENTS

Sold only by the Superintendent of Documents, Government Printing Office
Washington, D. C.

WASHINGTON
GOVERNMENT PRINTING OFFICE
1918

DETERMINATION OF PERMEABILITY OF BALLOON FABRICS

By Junius David Edwards

CONTENTS

	Page
1. Introduction	3
2. Theory of permeability process	4
3. Methods of determining permeability	5
(*a*) Volume-loss methods	6
(*b*) Penetration methods	7
4. Experimental apparatus	8
5. Effect of experimental conditions on apparent permeability	11
(*a*) Effect of temperature on permeability	12
(*b*) Effect of pressure on permeability	14
(*c*) Effect of humidity of hydrogen and air	15
(*d*) Effect of volatile substances in fabric	16
(*e*) Duration of test	18
(*f*) Rate of air flow	20
(*g*) Effect of size and shape of cell on apparent permeability	20
(*h*) Concentration of hydrogen	22
6. Volume-loss method	23
(*a*) Apparatus	23
(*b*) Results of tests	24
(*c*) Relation of volume-loss to penetration method	25
(*d*) Absorption of hydrogen by rubber	26
(*e*) Conclusions	27
7. Standard test	28
8. Operating directions and calculations	28
9. Accuracy of methods	30
10. Summary	31

1. INTRODUCTION

One of the most essential characteristics of a balloon envelope is that it shall be gas tight, or nearly so. Therefore the determination of the permeability of balloon fabrics to hydrogen, which is the gas used in the modern dirigible and kite balloons, is of first importance in determining their suitability for balloon construction. In connection with a study of the permeability of balloon fabrics it has been necessary to investigate different forms of apparatus and the influence of experimental conditions in order to interpret test results intelligently. The results of the

Bureau's investigation of methods of determining permeability are presented in this paper. The effect of composition, method of construction, etc., upon the permeability will not be discussed in the present paper, which deals only with testing methods.

A great many materials have been suggested for rendering fabric gas tight; the great majority of balloon envelopes, however, are made from rubber-coated silk or cotton, usually the latter. For the balloon envelope, two plies of cloth are used with the gas-tight rubber film between them. The two plies may be placed parallel to each other or one ply may be placed at an angle of 45 degrees; the biased fabric is supposed to offer greater resistance to tearing. Usually from 100 to 130 grams of rubber per square meter are placed between the plies. For protecting the fabric and the gas-retaining rubber film from the action of light, heat, moisture, etc., rubber coatings may be applied on the outside; further protection is sometimes added by coating the inside of the fabric also. Lighter single-ply fabrics are sometimes made for use in ballonets and three-ply fabrics find use in large dirigibles or supply balloons.

Goldbeater's skin finds some application in dirigible balloons of the rigid type. Oiled fabrics of various kinds, as well as fabrics coated with lacquers made from cellulose acetate or nitrate, have been tried. Various coatings have also been applied to rubberized fabrics with a view to rendering them more nearly gas tight and increasing their durability. However, none of these has been found entirely successful.

2. THEORY OF PERMEABILITY PROCESS

Gas escapes from a balloon envelope through leaks at valves, seams, and imperfections in the fabric, and by passage through the fabric. Hydrogen might pass through the fabric by diffusion through minute holes in a manner similar to the passage of a gas through a porous plate or it might escape by solution in the rubber film with subsequent evaporation from the other side. As a matter of fact, both processes occur simultaneously. The escape of hydrogen through openings in the fabric is only a case of gas effusion, but the passage of hydrogen through the rubber of the fabric is a very different phenomenon.

Thomas Graham,[1] in 1866, investigated the flow of gases through rubber films and showed that it was a solution phenomenon and

[1] Phil Mag., 32, p. 401; 1866.

not a diffusion phenomenon. If gases passed through rubber films by diffusion through microscopic holes, their relative rates of escape should be approximately inversely proportional to the square roots of their densities. Graham showed, however, that the rates of penetration of different gases bore no relation to their density. He found the relative rates of penetration of rubber by different gases to be as follows:

Nitrogen	1.0
Methane	2.15
Oxygen	2.56
Hydrogen	5.50
Carbon dioxide	13.58

The rate of change of permeability of rubber films with change of temperature is also much greater than can be accounted for by the diffusion theory. These and other facts have shown that gases penetrate rubber by first dissolving in the rubber.

When a rubber-coated balloon fabric is placed in contact with hydrogen on one side and air on the other, the following process is substantially what occurs. Both the hydrogen and the oxygen and nitrogen of the air dissolve in the rubber film and tend to saturate it up to the limits determined by the temperature and by the partial pressure of each gas. Each gas penetrates the rubber film at its characteristic speed, and when it reaches the opposite side of the rubber film the gas evaporates from its solution in the rubber because the partial pressure of the gas in question is substantially zero. More of each gas is dissolved continuously on the first side of the fabric and a dynamic equilibrium is reached which continues so long as fresh air and fresh hydrogen are available at the two surfaces. In this manner hydrogen passes through the rubber to the air side and air passes through to the hydrogen side.

These facts will be discussed more fully in connection with the experimental work and their bearing on methods of test pointed out.

3. METHODS OF DETERMINING PERMEABILITY

The permeability of a balloon fabric may be defined as the rate at which it is penetrated by a certain gas; except when stated otherwise, this gas is understood in this paper to be hydrogen. Permeability will be expressed in terms of liters of hydrogen per square meter per 24 hours, the volume of hydrogen being cor-

rected to the standard conditions of 0° C and 760 mm mercury pressure.

The methods in use for the determination of the permeability of balloon fabrics to hydrogen may be divided into two classes: (1) Methods which measure the rate of decrease in volume of a mass of hydrogen separated from the atmosphere by a definite area of the fabric; or (2) methods which measure by chemical or physical means the volume of hydrogen passing through a definite area of fabric in a given period.

The two methods may be characterized as the volume loss and penetration methods, respectively. According to the definition of permeability just given the volume-loss method does not measure the permeability of a fabric because in the volume-loss method the quantity measured is the volume of hydrogen escaping minus the volume of air passing into the hydrogen chamber.

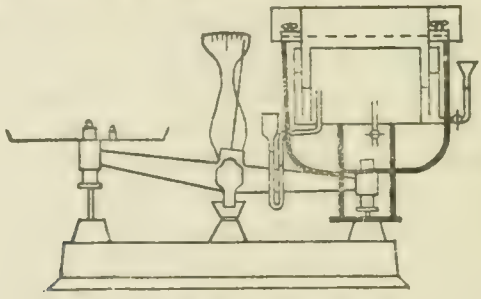


FIG. 1.—*Renard-Surcouf balance*

What it measures is the difference in the permeability of the fabric to hydrogen and air. Accordingly, any result obtained with this method will be characterized as the "volume-loss" permeability.

(*a*) *Volume-Loss Methods.*—The most widely used apparatus for applying the volume-loss method is known as the Renard-Surcouf balance. As shown in Fig. 1, it consists of the bell of a small gas holder mounted on one arm of a balance; the top of the bell is closed by the fabric (shown by a dotted line) under test. The bell moves up and down in an annular space filled with water; the annular container is attached to the base of the balance. The bell is first filled with hydrogen by alternately filling and emptying until the air has been displaced. The proper pressure is then obtained by adding weights to the opposite balance arm. As hydrogen escapes, the bell sinks, and the pointer moves over a calibrated scale indicating the volume loss. The apparatus should be kept in a constant temperature chamber.

Five other forms of apparatus of this class are described by Austerweil.[2] In each of these forms of apparatus a gas chamber filled with hydrogen is closed by the fabric under test. A graduated measuring tube is connected with this chamber and the volume of hydrogen escaping is measured by the volume of water necessary to replace it and maintain constant pressure. A leveling bottle may be used to introduce water into the measuring tube or it may be connected with a Mariotte flask which automatically introduces the water under constant pressure. If a leveling bottle is used, the pressure does not remain constant and must be adjusted at frequent intervals. The operating characteristics of this class of apparatus are discussed in section 6.

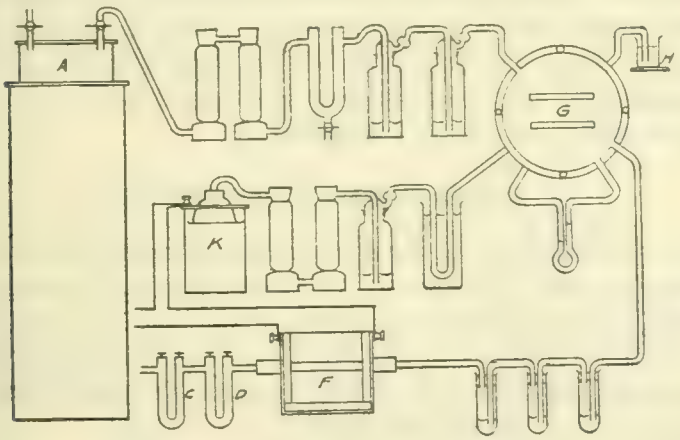


FIG. 2.—*Apparatus of Barr and Rosenhain*

(*b*) *Penetration Methods.*—The various forms of apparatus for applying the penetration method all employ a cell which is divided by the fabric into two chambers. In one of these chambers an atmosphere of hydrogen is maintained at a definite pressure. Through the other chamber is passed a stream of air which carries away the hydrogen as it penetrates the fabric. The amount of this hydrogen is determined by burning it to form water and weighing the water after absorption in some suitable medium, or the percentage of hydrogen is determined in the stream of air passing over the fabric by means of the gas interferometer.

The apparatus of Barr and Rosenhain,[3] as used in the National Physical Laboratory, is shown in Fig. 2. The hydrogen from the

[2] G. Austerweil, Die Angewandte Chemie in der Luftfahrt. Published by R. Oldenbourg, München, 1914.

[3] Report of the British Advisory Committee for Aeronautics, p. 86, 1909–10.

electrolytic cell *K* passes through a series of washing and drying tubes into the permeability cell *G* and out through the water seal *H*. The air from the gas holder *A* after passing through a series of washing and drying tubes passes over the other side of the fabric, then through another series of drying tubes, through an electric furnace *F*, where the hydrogen is burned, and finally through the absorption tubes *C* and *D* which are to be weighed. The permeability cell is kept in a constant temperature bath.

The apparatus described by Heyn[4] and used at the Physikalischen Reichsanstalt is similar in principle though differing in form and detail.

Frenzel[5] made a notable improvement in this method by employing a Rayleigh-Zeiss gas interferometer for determining the concentration of hydrogen in the air stream after passing over the fabric. From the percentage of hydrogen so determined and the rate at which the air was passing, as determined by a gas meter, the permeability was easily calculated.

4. EXPERIMENTAL APPARATUS

In the investigation of different testing methods several forms of apparatus were employed. The most useful apparatus and the one employed in all routine testing work as well as most of the experimental work is shown in Fig. 3. It applies the penetration method and employs a gas interferometer for determining the hydrogen passing through the fabric.

In this apparatus, the fabric is held in the premeability cell *a*, maintained at constant temperature in an air or water bath *h*. Pure, dry hydrogen passes over one side of the fabric and out through a water seal *g*.

Air after passing through the wet-pressure regulator *c* and a length of capillary tubing to reduce pressure fluctuations, is dried by bubbling through concentrated sulphuric acid in *e* and then passed over the other side of the fabric through a drying tube *d* [1] and into one chamber of the interferometer. It passes out through the drying tube *d* [2], which prevents diffusion of water vapor into the interferometer and then through the saturator *f* filled with glass beads partly covered with water and then through the wet meter *m*. The saturator is employed to prevent loss of water from the meter by evaporation into the gas being measured.

[4] Sitz. der Konig. Akad. Wiss., p. 160, 1911.

[5] "Über die Undurchlässigkeit der Gummierten Ballonstoffe." Zs. fur Flugtechnik und Motorluftschiffahrt, 5, p. 264, 1914.

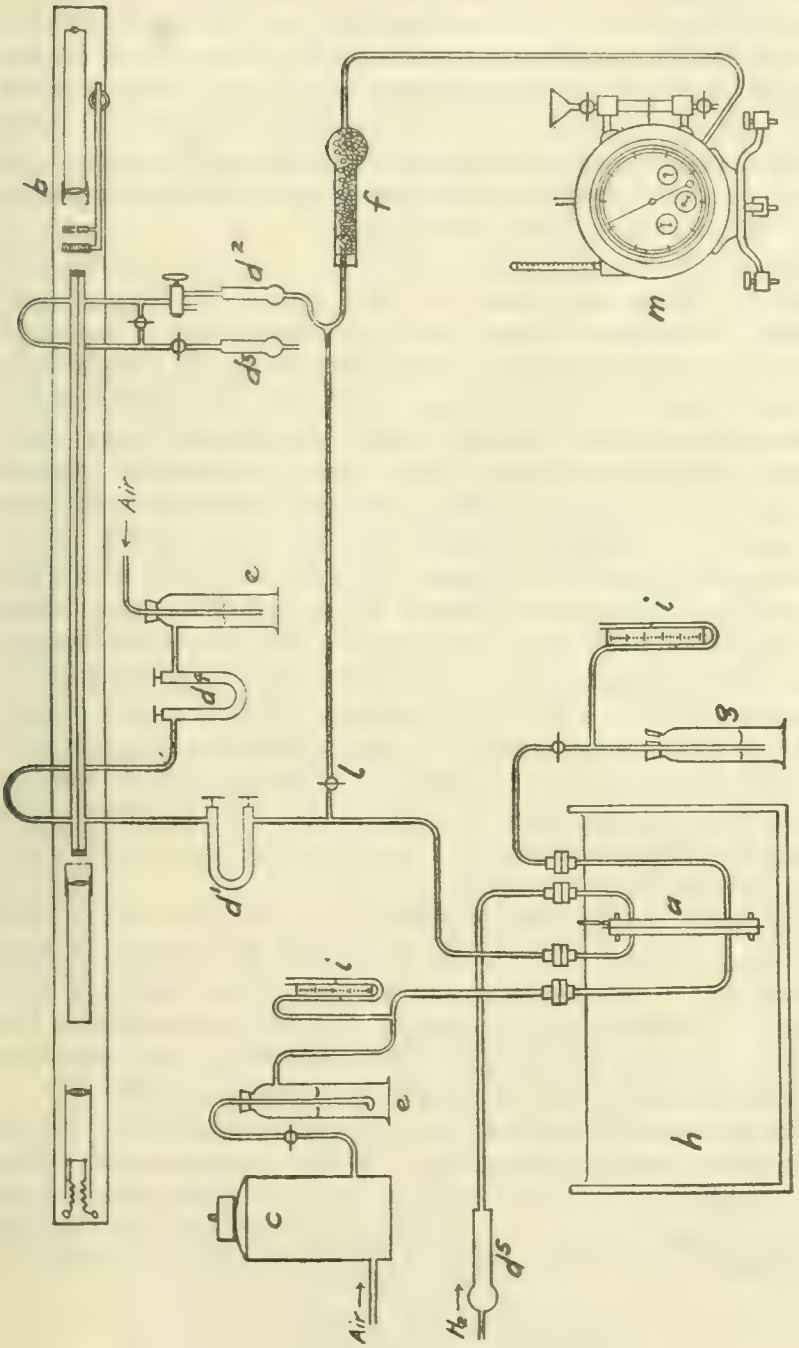


FIG. 3.—*Diagram of Bureau of Standards apparatus*

When the interferometer is being read, the air stream is by-passed through the cock *l* into the wet meter so as not to interrupt the flow of air through the permeability cell. The pressure on the air and hydrogen sides is measured by U-gages *i* filled with oil and water, respectively. Dry air is passed through the other chamber of the interferometer to serve as a comparison gas, or it may be passed through both chambers when it is desired to obtain the "zero reading" of the interferometer.

The gas interferometer and its uses have been described in detail by Haber and Lowe,[6] L. Stuckert,[7] L. H. Adams,[8] and others. The Rayleigh-Zeiss type of gas interferometer measures the difference in refractivity of two samples of gas contained in two gas chambers 100 cm long. Light from an illuminated slit passes through both chambers, after which the two beams combine to produce interference fringes which are observed through an eyepiece. The optical paths of the two beams can be brought to equality by tilting a glass compensator plate which is placed in the path of one of the beams. If the composition of the gas in one of the chambers is changed, the optical paths are different and the interference fringes are shifted. The fringes are brought back to their original position by tilting the compensator plate. The angle through which the compensator plate has been turned, which is measured by means of a drum attached to the micrometer screw, is a measure of the change in refractive index of the gas.

The instrument used for this work was of such sensitivity that one scale division indicated 0.01 per cent hydrogen in air when using air as a comparison gas.

The calibration of this instrument by a new method has been discussed by the author[9] in the Journal of the American Chemical Society. The interferometer furnishes a rapid and accurate method of determining the amount of hydrogen penetrating the fabric. Its many advantages will be pointed out in connection with the various subjects discussed in succeeding sections.

The permeability cells used are shown in greater detail in Fig. 4. The cell adopted as standard had a fabric area of 250 cm^2; other cells having areas of 1000, 100, 50, and 25 cm^2 were constructed for experimental purposes. In designing these cells the gas and air chambers were kept small in volume in order to reduce the

[6] Haber and Lowe, Zs. f. Angew. Chem., **23**, p. 1394: 1910.
[7] Stuckert, Zs. f. Elektrochemie, **16**, p. 37: 1910.
[8] L. H. Adams, J. Am. Chem. Soc., **37**, p. 1181: 1915.
[9] Edwards, J. Am. Chem. Soc., **39**, p. 2382: 1917.

space which must be swept out when operating. A large mesh-wire screen (3 cm openings) kept the fabric from being pressed against the back of the air chamber by the pressure of the hydrogen. The flanges of the cell contain a number of very fine grooves which take up the excess of grease used to make the fabric gas tight; they also give a better grip on the fabric. The two halves of the cell are held together by bolts and wing nuts (not shown in figure).

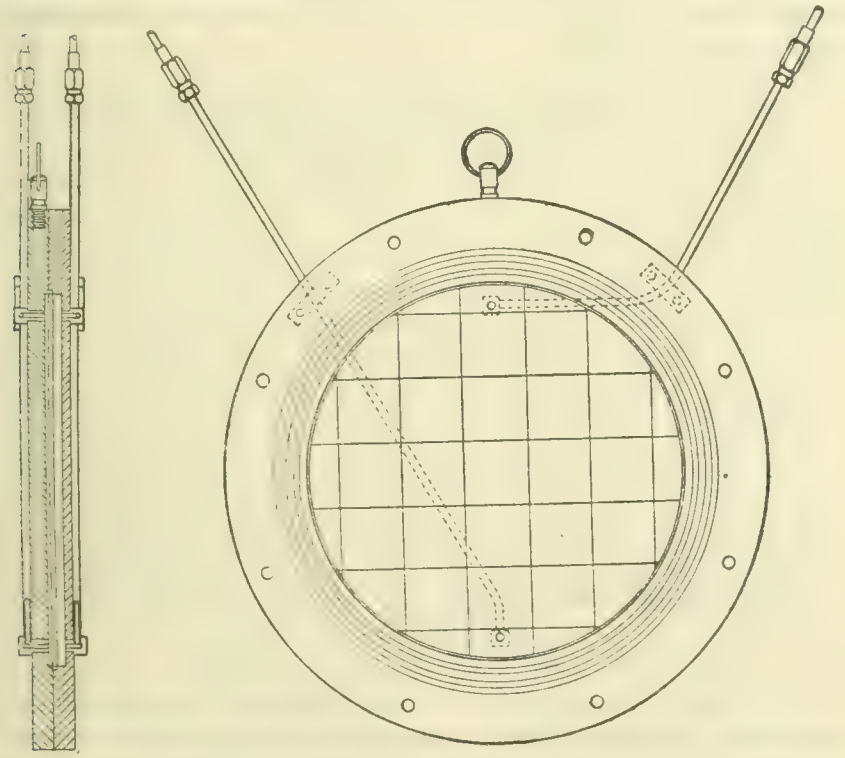


FIG. 4.—*Bureau of Standards permeability cell*

Connections to the gas and air lines and interferometer were made with small metal unions, and copper and glass tubing was used in making the connections to the various parts of the apparatus where necessary.

5. EFFECT OF EXPERIMENTAL CONDITIONS ON APPARENT PERMEABILITY

In order to interpret tests made under different conditions and and with different apparatus, the effect of experimental conditions on the apparent permeability was investigated. The appa-

ratus shown in Fig. 3, with various modifications, was used for all the work reported in this section. The general conclusions reached are applicable to all apparatus of this type whether the hydrogen is determined by combustion or by use of an interferometer; the operating details of the apparatus, however, may be quite different. The interferometer was used in preference to the combustion method because it greatly facilitated the testing work in point of speed and accuracy and permitted the carrying out of certain experiments which would otherwise have been impossible of execution.

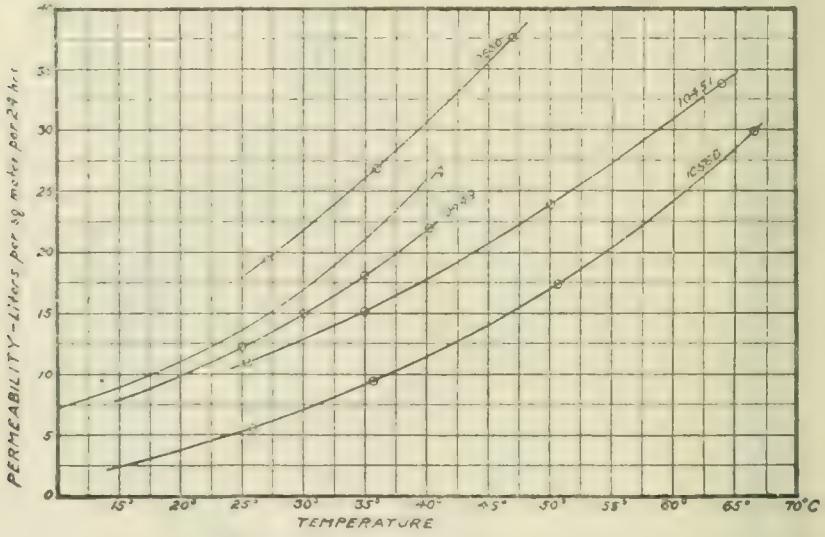


FIG. 5.—*Effect of temperature on permeability*

(*a*) *Effect of Temperature on Permeability.*—It has been known for a long time that the permeability of rubber films increases rapidly with increase of temperature.[10] For this reason it is necessary in all accurate work to maintain the fabric at a constant temperature during the period of test.

The rate of change of permeability with change of temperature for a number of different fabrics is shown in Fig. 5. In determining these curves the cell containing the fabric was mounted in an air bath, which was maintained at the desired temperature by means of an electric heater controlled by a thermostat. The fabric was allowed to come to equilibrium and its permeability determined at each temperature before changing

[10] Graham, Phil. Mag., 32, p. 421, 1866.

to the next temperature. Curves 10449, 10451, and 10650 show the temperature coefficients of fabrics from three American manufacturers and curve 10568 is for a fabric of foreign manufacture. These curves are all very similar in shape, indicating that, approximately at least, the same relation between permeability and temperature holds for fabrics of quite different composition. For purposes of comparison, another curve showing the change of permeability with temperature as determined by other experimenters is included. This curve *A* is taken from the report of Gibbons and Smith;[11] it shows substantial agreement with the Bureau's results. Frenzel[12] gives graphs for four continental balloon fabrics. They show a smaller rate of change than any of the preceding curves and indicate the change of permeability to be a linear function. None of the fabrics examined by this Bureau have shown a linear relation between temperature and permeability. Communications from private testing laboratories here and abroad show temperature effects corresponding to those obtained at the Bureau and do not indicate a linear relation. Two tests at two different temperatures by Rosenhain and Barr[13] indicate a somewhat more rapid increase of permeability with increase of temperature. Their data are insufficient to warrant any general conclusions, however.

In order to make the tests comparable, the Bureau of Standards has adopted the practice of making all tests at a standard temperature of 25° C. This temperature was adopted because of the ease and convenience with which the testing apparatus could be maintained at this temperature, and because it was thought to represent with some degree of approximation an average balloon temperature.

The National Physical Laboratory, England, has adopted 15.5° C (60° F) as its standard temperature. That temperature can not be maintained in most American testing laboratories in summer without refrigeration, which needlessly complicates the testing. The customary practice in France is to use 15° C as the standard temperature. However, temperature control of the Renard-Surcouf balance, which is the most widely used instrument there, is rather difficult, and it is not known what degree of uniformity there is in temperature control in French practice.

[11] First Annual Report of the National Advisory Committee for Aeronautics (Washington, [illegible]).

[12] Frenzel, "Über die Gasdurchlässigkeit der Ballonstoffe." Druckerei des Elsassichen Textilblattes im Gebweiler.

[13] Report, Great Britain Committee on Aeronautics, p. 60; 1910–11.

For the purpose of approximate comparisons, the permeability of the average rubberized fabric at 15° C may be taken as 65 per cent of the permeability at 25° C.

(b) *Effect of Pressure on Permeability.*—It has been customary, both here and abroad, to determine the permeability of balloon

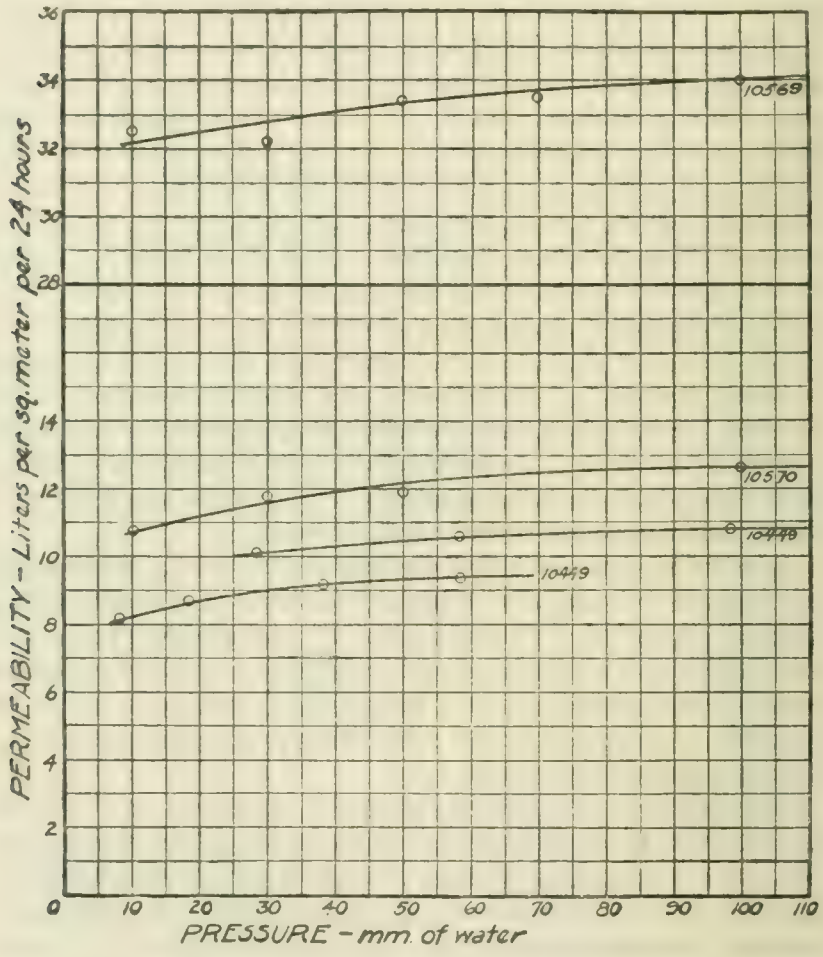


FIG. 6.—*Effect of pressure on permeability*

fabrics under a pressure of hydrogen equivalent to 30 mm of water. This is approximately the normal working pressure of a nonrigid balloon.

The pressure coefficient for two-ply balloon fabrics has been found to be rather small. The rate of change of permeability with change of hydrogen pressure for three fabrics is shown in Fig. 6.

The change in permeability with change in pressure is very small, above 50 or 60 mm the curve being almost horizontal. The greater effect of pressure change at low pressures may be due, as suggested by Frenzel, to the fact that the initial increases of pressure stretch both the rubber film and the supporting fabric, but the point is soon reached where further stretch of the rubber film is prevented by the relatively inelastic fabric, and thereafter the permeability shows only a very small change. The effect of pressure as well as of temperature varies somewhat with different fabrics, but with those tested it was small. This is to be expected, since an increase of hydrogen pressure of 10 mm of water (0.74 mm of mercury) means an increase of only about 0.1 per cent in the total pressure of the hydrogen.

Since the permeability is approximately proportional to the partial pressure of hydrogen (see p. 22), the permeability should vary with the atmospheric pressure, because the partial pressure of the hydrogen is equal to the atmospheric pressure plus the constant excess pressure under which it is confined in the cell. However, the changes in barometric pressure are usually small, seldom more than 1.5 per cent; accordingly, they have been neglected in routine testing since their effect upon permeability, as far as can be ascertained from an examination of test results, is within the limits of experimental error. It should be noted that it is the excess gas pressure above atmospheric which produces the greatest part of the increase noted at the beginning of the curves shown in Fig. 6.

(*c*) *Effect of Humidity of Hydrogen and Air.*—The influence of moisture upon the permeability was determined by comparing the permeability shown when using dry hydrogen and air with the permeability when using moist hydrogen and air. When dry gas was to be used, it was passed through concentrated sulphuric acid; when moist gas was employed, it was passed through sulphuric acid of such concentration that the relative humidity of the saturated gas was approximately 65 per cent at 25° C.

The permeability of a fabric was determined with dry gas and then with moist gas; the order was then reversed. The same difference was noted in each case when testing under the two different conditions. The average results of these tests are given in Table 1.

TABLE 1.—Effect of Humidity of Hydrogen and Air on Permeability

Order of test	Condition of hydrogen and air	Permeability, liters per square meter per 24 hours	Difference	Order of test	Condition of hydrogen and air	Permeability, liters per square meter per 24 hours	Difference
First test	Wet	13.8		First test	Wet	13.7	
Second test	Dry	14.4	0.6	Second test	Dry	14.5	0.8
First test	do	15.1		First test	do	14.6	
Second test	Wet	14.5	.6	Second test	Wet	13.9	.7

When the hydrogen is 65 per cent saturated at 25° C there is approximately a 2 per cent decrease in the partial pressure of the hydrogen due to the addition of water vapor. This partially explains why a 4 to 5 per cent decrease in permeability was noted when changing from dry gas to wet gas. The amount of water dissolved in the rubber probably also influences the rate of penetration of the rubber by the hydrogen.

Tests showed that the moisture contained in the fabric was not completely removed by a current of dry air at 25° C after a number of hours' passage, so that it is certain that the rubber compounds under the conditions of test with dry gas still contain an appreciable amount of water vapor.

No further experiments on the effect of moisture were made, since it was evident that the difference in permeability under the two conditions of test was small—less than 1 liter in this case. For testing purposes the conditions of test which are recommended should be easily obtained and duplicated, and it is obvious that the use of dry hydrogen and air is most satisfactory in every respect. It does not introduce further complications in calculations and testing, and gives results very close to those obtained with partially saturated gas.

(*d*) *Effect of Volatile Substances in Fabric.*—The apparent permeability may be affected by the removal of volatile substances from the fabric by the current of air passing over it. There may be present in the air stream after passing over the fabric varying amounts of water vapor, carbon dioxide, or the vapor of benzol, gasoline, or other solvent used in the manufacture, in addition to the hydrogen which has penetrated the fabric. The water vapor and carbon dioxide which may have been dissolved in the rubber are removed by soda lime and calcium chloride before the air

stream enters the interferometer. In case the combustion method is used, only the water vapor need be absorbed.

The presence of vapor of solvents, however, introduces a possible serious source of error into either method. If the hydrogen is estimated by use of the interferometer, the apparent permeability is too low, because the refractivity of solvent naphtha or benzol is higher than the refractivity of air or hydrogen. If the combustion method is used, the results are too high by the amount of water formed by the combustion of the solvent. This point should be carefully considered when testing fabrics of very recent manufacture, and it is of great importance in the commercial control of fabric manufacture. It is usually assumed that the solvent used in the rubber coatings when they are spread is entirely removed by the subsequent heating and curing. In some cases, notably three-ply fabrics with heavy rubber coatings, sufficient solvent may be left to seriously vitiate test results unless it is removed or corrected for. Its presence can sometimes be detected from the odor of the fabric.

The necessary correction for solvent vapor can be readily found by determining the reading of the interferometer when using air in place of hydrogen in the permeability cell. Since the amount of solvent given off by the fabric decreases with time, it is necessary to take at least three readings at short intervals to determine the rate of change of the blank; the correction at the time of the permeability test, which should follow immediately, is estimated by extrapolation. This method does not necessitate waiting for the complete removal of the solvent before beginning the test, and for most purposes is sufficiently accurate. It is of course obvious that a more accurate method would be to wait until the solvent is completely removed, thus eliminating the necessity for a blank correction.

The estimation of the blank correction is more difficult in case the combustion method is used, because it is impracticable to determine its rate of change. Its magnitude may be determined, however, and if a more accurate determination is required, a number of permeability tests can be made at suitable intervals.

Of several hundred samples tested at the Bureau only fabrics of a certain composition have shown blanks of appreciable magnitude. The possibility should not be overlooked, however, in the case of any fabric with heavy rubber coating and of very recent

manufacture, particularly if the presence of solvent can be detected by the odor.

(c) *Duration of Test.*—The passage of hydrogen through the fabric being a solubility phenomenon, it may be assumed that a certain time must elapse after the fabric is placed in contact with hydrogen before the fabric is saturated with hydrogen and reaches its maximum permeability. The interferometer lends itself readily to the investigation of this point, since readings may be taken at short intervals.

Care should be taken not to confuse the time required for the air-hydrogen mixture to reach equilibrium throughout the apparatus with the time required for the fabric to reach equilibrium. The larger the air chamber in the cell and the greater the volume of the rest of the air system, the longer is the time required for the hydrogen given off from the fabric to reach a uniform concentration throughout the apparatus. It was with this point in mind that the cells were designed with as small gas chambers as seemed practicable.

The results of 200 representative determinations were examined to ascertain what effect the duration of the test had upon the apparent permeability. In making these determinations, which were mainly of a routine nature, the first reading was usually taken 30 minutes after starting the test. Three or four readings at intervals of approximately 30 minutes were taken in order to determine whether a constant value was being reached. These results, together with the experience obtained in some 500 other tests, show that the time required for the fabric to reach its maximum permeability is almost always less than 1 hour and usually less than 30 minutes.

Other workers have stated that periods of from 3 to 5 hours were necessary before the fabric reached its maximum permeability. Where experiment seemed to indicate this, the time was probably required for the air stream itself to reach equilibrium and not the fabric. For any apparatus the preliminary sweeping-out period should be experimentally determined.

It has been noted that in continued testing of the same piece from day to day there was usually a gradual decrease in the permeability. For example, a certain test piece (No. 52), which had an initial permeability of 15.1 liters, showed on the 3 succeeding days permeabilities of 14.8, 14.6, and 14.4 liters. Sometimes the decrease was more rapid; another fabric (test No. 518), showed a

decrease from 21.4 to 19.2 liters in 5 successive half-hour tests. One very unusual 3-ply fabric with extra heavy rubber coatings consistently showed a large decrease; 1 test piece, for example, changed in permeability from 18.9 to 14.5 liters in 8 successive tests on the same day. The same test piece changed from 16.4 to 11.3 liters on the following day. Four months later another test piece from the same sample indicated in 5 successive half-hour tests permeabilities of 17.2, 16.1, 14, 13.6, and 12 liters. However, the great majority of fabrics tested give quite consistent and constant results during the period of test. Using the combustion method, where a considerable period of time is necessary for each test and usually only one observation is made, the changes noted above would usually pass unobserved.

A similar decrease in permeability is obtained by heating the fabric. Test piece No. 52, previously referred to, showed a permeability of 14.4 liters at the last test. Without removing it from the cell or interrupting the test its temperature was raised to 70° C and held at that temperature for 30 minutes. On lowering the temperature to 25° again its permeability was found to be 10.8 liters, and on the 3 succeeding days it was 10.9, 10.9, and 11 liters. Another test piece showing an initial permeability of 11.7 liters was heated to 50°, held at that temperature for 30 minutes, and then allowed to cool again, the whole process requiring about 3 hours. Its permeability was found to have decreased to 8.5 liters. Fabric heated in an electric oven before testing and before coming in contact with hydrogen showed a similar decrease in permeability, the magnitude of the effect depending upon the temperature and time of heating. Samples exposed to the weather also gradually acquire a lower permeability. This, however, is accompanied by a hardening process, which eventually leaves the rubber film so brittle as to be useless.

This variation in the permeability of a rubber film under the conditions noted makes it impossible to use one test piece in a long series of experiments and yet secure comparable results. New pieces must be used to secure comparable samples, and they are subject to any variation due to nonuniformity of the material.

In conclusion, it may be said that the permeability of most samples remains practically constant during a day's test. Longer testing will probably show a decrease. Occasionally a fabric will be met with that shows a very large decrease during the test. Heating a fabric will produce a very appreciable decrease in per-

meability. An explanation of the changes occurring in the rubber which cause the changes in permeability noted will not be given at this time.

(*f*) *Rate of Air Flow.*—Tests made over an extended period with different fabrics and different permeability cells showed that, within wide limits, changing the rate of passage of air through the cell did not make any significant change in the apparent permeability. The air rate in both large (250 cm²) and small (100 cm²) cells was varied from 1 to 3 liters per hour; the normal rates were 1.5 and 2.5 liters per hour. The tendency toward slightly higher results when the rate was increased was occasionally noted, but the effect was not consistently observed; usually any change was well within the accidental variations in the results. It may be concluded, therefore, that within working limits, differences in the air rate are unimportant as affecting the result. It should be noted, however, that a uniform air rate must be maintained during the test in order to secure satisfactory results. This is especially important where the interferometer is being used, since it is assumed that the concentration at the time of observation is the same as during the preceding period when the rate was being determined by the meter; this would only be true in case the permeability and rate of air flow remained constant

(*g*) *Effect of Size and Shape of Cell on Apparent Permeability.*—The size and form of the cell might have an effect on the apparent permeability of a fabric being tested in it if the cell did not permit uniform circulation of the hydrogen and air currents over the entire area of the test piece. If the air is not readily swept out of the hydrogen chamber or if the hydrogen passing through the fabric is not uniformly removed by the air stream, the results with different cells might vary. The significant factors of cell design have been examined to determine what effect they have upon the apparent permeability.

The first point considered in the design of the cell was the size of the test piece and the form of the hydrogen and air chambers. The method depends of course upon reaching an equilibrium condition throughout the system with practically pure hydrogen on one side of the fabric and a uniform distribution throughout the interferometer system of the hydrogen passing through the fabric. Therefore, the smaller the volume of the gas chambers in the cell, the sooner will this equilibrium condition be reached, providing, of course, that the fabric is being penetrated by hydrogen at its

maximum rate under the given conditions. On the other hand, the area of the test piece must be large enough to give sufficient concentration of hydrogen at a suitable air rate for the required accuracy in the interferometer reading.

For most work a test piece having an area of 250 cm² is satisfactory. The construction of the cell adopted as standard is shown in Fig. 4. The hydrogen chamber has a depth of 2 mm; the pressure of hydrogen (30 mm of water) is sufficient to keep the fabric from coming in contact with the cell wall. The air chamber is 4 mm deep; to prevent the pressure of the hydrogen from forcing the fabric against the opposite side of the chamber a large-mesh wire screen is placed in the cell. Three cells of this type and a fourth exactly similar, except that the air chamber is 6 mm deep, are in constant use. Intercomparison shows that all four give practically identical results with the same fabric. Equilibrium is reached very quickly with a cell of this type. (See p. 18.)

A smaller cell having an area of 100 cm² and a hydrogen chamber 20 mm deep was modified by filling up the hydrogen chamber with paraffin until the gas space was only 2 to 3 mm deep. With the original hydrogen chamber of 20 mm depth the fabric did not reach its maximum permeability until several hours after starting the test, even though the rate of passage of the hydrogen was several times the normal rate. With the hydrogen space restricted a constant permeability was reached 30 minutes after starting the test.

The cells are normally used in a vertical position. To determine whether this had any effect on the result, a cell was operated in various other positions. As was to be expected, no effect due to position was observed.

In a similar manner the effect of direction of gas flow was tested. With a cell suspended in a vertical position it made no difference in the apparent permeability whether the air was introduced at the top or at the bottom of the cell. In operation it is our practice to introduce the hydrogen into the cell at the top and the air at the bottom, on the theory that the cell will be swept out more rapidly. It probably makes little, if any, difference, however, if the chambers are small and the gas current sufficiently rapid.

To determine what effect the area of the test piece had on the apparent permeability, cells of different sizes were constructed. These cells had fabric areas of 50, 100, 250, and 1000 cm².

If the hydrogen which passes through the fabric is not rapidly removed from the surface, the partial pressure of the hydrogen might become sufficiently great to lower the apparent permeability appreciably. Any such increase of the partial pressure of hydrogen at the surface of the fabric is prevented by the high rate of diffusion of hydrogen. The accumulation of hydrogen in the cell is prevented by the current of air which reduces the average concentration of hydrogen in the cell to 1 per cent or less. For these reasons it is to be expected that the apparent permeability would be independent of the rate of air flow and of the area of exposed fabric within rather wide limits.

The results of a series of tests with these cells showed that these conclusions were substantially correct. The small (50 cm^2) cell gave practically the same apparent permeability as the 250 cm^2 cells but the results were less concordant. The agreement between the 100 and 250 cm^2 cells was very good. With the 1000 cm^2 cell apparently a longer time was required to reach equilibrium.

If the hydrogen is able to penetrate laterally between the plies (see footnote 15, p. 25) the effective area of exposed fabric may not be defined by the edge of the gas chamber but may be somewhat larger. This edge effect would of course be proportionally greater in a small cell than in a large one. However, no such consistent difference was noted.

(*h*) *Concentration of Hydrogen.*—The tests discussed so far were made with hydrogen of approximately 100 per cent purity. If the concentration of the hydrogen in contact with the fabric be reduced by admixture of air, it seems probable that the apparent permeability of the fabric to hydrogen will be reduced about proportionally. Barr and Thomas [14] conclude from two experiments with mixtures of air and hydrogen that the permeability is directly proportional to the partial pressure of the hydrogen.

No tests of this point have been made as yet at the Bureau, since for testing purposes the use of pure hydrogen is desirable from every standpoint and it is known that if the hydrogen contains air, the apparent permeability to hydrogen will be reduced about proportionally. This is an important point to consider in connection with volume-loss methods, since the diffusion of air into the hydrogen chamber will lower the partial pressure of the hydrogen and consequently the apparent permeability.

[14] Report Great Britain Committee on Aeronautics, 4, p. 276; 1912.

6. VOLUME-LOSS METHOD

Because of the comparative simplicity of the volume-loss method it is important to determine the precision and reliability of results obtained with that method. In the discussion of the theory of the permeability process it was stated that the penetration of the fabric by the hydrogen was accompanied by a simultaneous passage of air through the fabric in the opposite direction. For this reason the volume loss determined is not the hydrogen which has escaped but that volume less the volume of air passing through the fabric into the hydrogen chamber. It is of interest to know what the numerical relation between results determined by the two methods is; also, if there is any great variation in the relative rates of penetration of rubber films by hydrogen and air, it is a fact of importance.

FIG. 7.—*Volume loss apparatus*

(*a*) *Apparatus.*—For investigating the volume-loss method, the apparatus shown in Fig. 7 was designed. It consists of a cell similar to that shown in Fig. 4 and identical with the cell of that type having an area of 100 cm^2 except that the hydrogen chamber is considerably larger (volume = 275 cm^3).

The large volume of the hydrogen chamber prevents any considerable reduction of the partial pressure of the hydrogen through the small volumes of air which diffuse inward. Attached to the hydrogen chamber is a graduated measuring tube which is connected by the orifice shown near the top with a constant-pressure flask. As hydrogen escapes through the fabric, water from the constant-pressure flask drops into the burette through the orifice to take its place. The decrease in volume of the confined gas is determined by the increase of volume of water in the burette. The total volume of the gas space is known and corrections are applied for the changes in temperature and pressure which may occur during the test. Since the fabric

changes temperature with the surroundings, a further correction is necessary for the change of permeability of the fabric with change of temperature.

In operation, the fabric was clamped tightly between the well-greased flanges of the cell. The gas chamber was then tested for leaks by placing it under a small pressure of air. If it was air-tight, the air in the gas chamber and burette were swept out with hydrogen, and the chamber left filled with hydrogen under a pressure of 30 mm of water. The decrease in gas volume was then noted at 30-minute intervals. A current of air was passed through the upper chamber during the period of test.

This apparatus is similar in principle to the apparatus of Lebaudy, Sabbatier, and others and combines their best features. It was developed primarily for experimental purposes.

(*b*) *Results of Tests.*—The results obtained with this apparatus, using a series of fabrics from three different manufacturers, are shown in Table 2. For purposes of comparison the permeabilities of the same fabrics as determined with the apparatus shown in Fig. 3 are included.

The conclusions drawn from these results and the experience in the use of this and similar apparatus may be summarized as follows:

TABLE 2.—Apparent Permeability as Determined by the Volume-Loss Method

Fabric No.	Apparent permeability at 25° C by volume-loss method, liters per square meter per 24 hours	Permeability at 25° C by penetration method, liters per square meter per 24 hours	Ratio of permeabilities	Fabric No.	Apparent permeability at 25° C by volume-loss method, liters per square meter per 24 hours	Permeability at 25° C by penetration method, liters per square meter per 24 hours	Ratio of permeabilities
22152	10.9	15.8	0.69	10652	14.4	20.1	0.72
22151	11.0	15.8	.70	22795	9.8	14.4	.68
22150	12.0	17.9	.67	22496	9.9	15.0	.66
10449	10.7	14.5	.74				
10650	12.4	17.1	.72				Av. .69
10651	7.2	11.0	.65				

With this size of cell the actual volume of hydrogen escaping per hour is usually small and the errors of measurement relatively large. The results are not very concordant and the precision secured is low compared with the standard apparatus.

Great care must be taken to secure a gas-tight joint between the fabric and the cell in order to prevent any leakage other than

through the fabric. A small leak in the hydrogen chamber of apparatus of the penetration type is unimportant, but with the volume-loss method it is fatal to accuracy. Even if this joint is gas-tight, hydrogen may escape from the cell by leaking out between the plies of the fabric. Excessive permeabilities indicated in several cases were traced to this source.[15] To guard against this, if possible, the edges of the fabric were coated with shellac. Where a high permeability is indicated, the results are usually open to the suspicion of being influenced by a leak.

(*c*) *Relation of Volume-Loss to Penetration Method.*—To determine the theoretical relation between the volume-loss and penetration methods it is necessary to know the relative permeability of balloon fabrics to air and to hydrogen.

According to the figures given by Graham (p. 5) the relative rates of penetration of rubber by nitrogen, oxygen, and hydrogen are 1, 2.56, and 5.5. Since the permeability is proportional to the partial pressure, the relative permeability to hydrogen and air would be as $\frac{5.5}{(0.79\times 1+0.21\times 2.56)}$, which is as 4.1 is to 1. These figures, of course, will vary with different rubber compounds and different experimental conditions.

The rates at which hydrogen and air penetrate a balloon fabric have been determined simultaneously by using two interferometers. With the standard cell (Fig. 4) the hydrogen passing into the air stream was determined in the usual manner. The air passing into the hydrogen in the other chamber of the cell was determined at the same time in another interferometer by comparison with hydrogen from the same source which had not passed through the cell. The calibration of the interferometer was somewhat in doubt in this latter case owing to the fact that the "air" in the hydrogen may have a different ratio of oxygen to nitrogen[16] than in atmospheric air (see p. 5), but if the oxygen content was as high as 40 per cent, the readings would be approximately only 3 per cent low. This difference falls within the rather large experimental uncertainty in these tests; accordingly no correction has been made. This was allowable since the ratio to be determined is not a constant and the results are sufficiently concordant for present purposes.

[15] Fabric No. 10653 consistently permitted gas to leak between the plies of the fabric and out at the edge of the test piece.

[16] "Air" after passing through a rubber film usually contains more than 21 per cent oxygen because of the higher permeability of rubber to oxygen than to nitrogen.

TABLE 3.—The Permeability of Balloon Fabrics to Hydrogen and Air

Fabric No.	Permeability to hydrogen, liters per square meter per 24 hours	Permeability to air, liters per square meter per 24 hours	Ratio hydrogen to air rate
19652	29.5	8.4	3.5
24[illegible]5	17.6	4.4	4.0
24[illegible]3	14.2	3.8	3.7
24[illegible]3	5.4	1.4	3.9
			Av. 3.8

Table 3 shows the results for four different fabrics. The average ratio of hydrogen to air is about 3.8 to 1. This ratio seems to be approximately constant for rubber-coated balloon fabrics of the type now in use. This would indicate that the volume-loss method should give results which are approximately 74 per cent $\left(\frac{2.8}{3.8}\right)$ of the penetration method, since while 3.8 liters of hydrogen are escaping through the fabric, 1 liter of air passes into the gas chamber, making a net loss of 2.8 liters. According to Graham's figures, which were determined under very different experimental conditions and with different rubber compounds, the volume loss would be $\left(\frac{3.1}{4.1}\right)$ or 76 per cent[17] of the permeability determined by the penetration method; this ratio is very close to the one found by the author. The volume-loss results given in the table were on the average 69 per cent of the values secured with the same fabrics by the penetration method. The difference between 69 per cent and the value (74 per cent) just calculated may be attributed to the fact that the hydrogen was saturated with water vapor in the volume-loss experiments and was used dry in the other method. It was shown on page 16 that the apparent permeability was 4 to 5 per cent lower when moist gas was used.

(d) Absorption of Hydrogen by Rubber.—In connection with the tests of the apparatus shown in Fig. 7 a series of determinations were made of the rate at which hydrogen is absorbed by the rubber in a balloon fabric. The tests were made by placing a fabric in the apparatus as for a determination of permeability, and then cutting slits in the fabric so as to connect both chambers of the cell. Both chambers were then filled with hydrogen, closed, and the rate

[17] Austerweil (Die angewandte Chemie in der Luftfahrt, p. 58), by erroneous reasoning, arrives at the figure 71 per cent for this relation.

at which the hydrogen was absorbed was measured in the usual way. The hydrogen absorbed was calculated in terms of liters per square meter per 24 hours. The results are shown in Table 4. A test on the cloth without the rubber coating showed no absorption of hydrogen, thus proving what was evident—that the hydrogen was absorbed by the rubber and not by the cloth.

TABLE 4.—Absorption of Hydrogen by Rubber-Coated Balloon Fabric

Fabric No.	Permeability, liters per square meter per 24 hours	Rate of absorption of hydrogen, liters per square meter per 24 hours						Milligrams of hydrogen absorbed per gram, rubber compound
		First 30 minutes	Second 30 minutes	Third 30 minutes	Fourth 30 minutes	Fifth 30 minutes	Sixth 30 minutes	
22496	15.0	4.3	.. ....	1.4	0.0	0.0	... .	0.08
22150	17.9	3.4	1.0	...	.5	.0	0.0	.09
22151	15.8	1.0	1.0	.5	.0	.0	.0	.04
22152	15.8	2.4	1.0	. .	.0	.0	.....	.04
10651	11.0	.0	.	.0	.....	...	.....	.....
10652	20.1	.. .	1.0		2.4	.. ..	.0	.08

These experiments indicate that saturation of the rubber with hydrogen is practically complete in 60 to 90 minutes. Kayser [18] also found, using another method, that absorption was complete in about 1 hour; no further absorption was noted by him in experiments lasting 3 to 5 days. In the determinations by the volume-loss method shown in Table 2 the reading at the end of the first 30-minute period was usually about a liter higher than the average of the 8 to 10 readings taken. Although the data are not very complete, it seems evident that a higher initial reading might be partly due to this absorption of hydrogen by the rubber. It does not, however, make a significant difference in the results because of the rather large accidental errors involved in the volume-loss measurements.

(*e*) *Conclusions.*—It may be concluded from the results of these tests and many others unrecorded and from the experience of other experimenters as noted in the literature that all volume-loss methods so far developed are inferior to the penetration method both in precision and reliability. The permeability according to the volume-loss method is approximately 70 per cent of the permeability by the penetration method.

[18] Wied. Annalen der Phys. und Chem., 43, p. 547; 1891.

7. STANDARD TEST

The permeability test adopted as standard by the Bureau of Standards may be briefly described as follows: The fabric to be tested is held in a permeability cell of the size and form shown in Fig. 4, or its equivalent, and is maintained during the period of test at a temperature of 25° C. A current of pure dry hydrogen is passed over one side of the fabric under a pressure of 30 mm of water above the pressure on the other side of the fabric. Dry air, at approximately atmospheric pressure, is passed at a constant rate through the other chamber of the cell and the hydrogen passing through the fabric into the air is determined by means of a gas interferometer and meter. The hydrogen may also be determined by combustion and weighing as water. If the interferometer is used, a reading is taken every half hour until a constant permeability is indicated. If the combustion method is used, the fabric should remain in the apparatus in contact with the atmosphere of pure hydrogen for a period of one hour before beginning a test.

The permeability is calculated in liters of dry hydrogen escaping per square meter of fabric per 24 hours, the volume of hydrogen being corrected to the standard conditions of 0° C and 760 mm pressure of mercury.

8. OPERATING DIRECTIONS AND CALCULATIONS

In the preceding sections many of the details of operation such as pressure, temperature, etc., have been discussed at length. It may be well to call attention also to certain other points which must be taken account of in order to secure concordant results.

To begin with, the fabric should be firmly fastened in the cell and the area of fabric exposed to the hydrogen should be definitely known. If the faces of the cell are not exactly plane or if they are insufficiently lubricated or if the cell is not bolted together tightly, hydrogen may leak past the edge of the gas chamber and increase the area of the exposed fabric. The results will accordingly be too high and will probably be erratic.

The air current should be maintained as uniform as possible. The air can be forced through under low pressure or it may be drawn through by slightly reducing the pressure at the outlet with a water pump or other vacuum pump. To aid in maintaining a uniform pressure, a water-sealed, gas-pressure regulator may

be used. A long piece of capillary glass tubing inserted in the air line also tends to reduce fluctuations in the rate of air flow.

The hydrogen should be passed through the cell rapidly at the start of a test in order to sweep out the air as quickly as possible. When the air is removed the hydrogen need be passed only slowly in order to sweep out the air which diffuses through the fabric.

The permeability is calculated from the following equation:

$$P = \frac{S \times F \times H}{A}$$

P = permeability in liters per square meter per 24 hours.
S = rate of air passage, liters per 24 hours.
A = area of fabric in square meters.
F = factor by which indicated air rate must be multiplied to reduce the gas volume from the condition of saturation at the temperature and pressure in the meter to the volume when dry and at 0° C and 760 mm pressure.
H = percentage of hydrogen in the air stream.

The factor F is calculated as follows:

$$F = \frac{p - p_w}{760} \cdot \frac{273}{t + 273}$$

p = barometric pressure plus pressure above atmospheric in the meter.
t = temperature of meter.
p_w = vapor pressure of water at temperature t.

A table giving the value of F for different temperatures and pressures should be prepared if many tests are to be made.

Most of the considerations discussed in section 5 apply equally well to a method in which the hydrogen is determined by combustion with subsequent weighing as water instead of by means of an interferometer. The advantages the interferometer offers in point of speed and precision have already been pointed out. If a suitable interferometer is not available, however, the hydrogen may be determined by combustion; in fact, this is the customary method in commercial use.

To secure correct results by combustion, it is necessary that the air and hydrogen from the cell be perfectly dried; that the hydrogen be completely burned; that the water formed be completely absorbed and its weight correctly determined. This can be done with sufficient accuracy if the proper precautions are taken.

The efficiency of drying with any apparatus and drying agent should be tested by blank runs. It is desirable to use two absorption tubes in series in order to determine the efficiency of drying in the first tube. When the second drying tube begins to absorb increasing amounts of moisture, the first tube should be refilled.

The combustion of the hydrogen may be accomplished in a number of ways, such as by passing over heated copper oxide, platinized asbestos, platinized quartz, palladium black, glowing platinum wires, etc. Complete combustion can be secured by any of these catalysts if the proper temperature is employed and the gas is not passed through the combustion tube too rapidly. In any case it is well to determine by experiment that the combustion is complete under the conditions of use. It is important that the rate of passage of the air be not too rapid to prevent efficient drying and complete combustion.

Since the amount of water collected is usually small, every precaution should be taken in weighing the glass absorption tubes. It is desirable that a counterpoise of as nearly the same size and shape as the absorption tube be used in weighing. The absorption tubes should be wiped with a damp cloth and hung in the balance case or a suitable container to permit them to assume the temperature of the balance before weighing. Rubbing the glass while dry is likely to produce electrostatic charges on the glass which prevent accurate weighing.

9. ACCURACY OF METHODS

The accuracy with which the permeability of a fabric can be determined depends upon the accuracy with which the various factors of the test, such as concentration of hydrogen, etc., can be determined, and is closely associated with the question of uniformity of fabric. Some fabrics show very close agreement on duplicate tests and give evidence of being very uniform. Fabric No. 24579 is a good example of this. Tests on eight different test pieces of this fabric showed permeabilities between 8.6 and 8.9 liters. Experience with this fabric indicates that a great many of the differences noted between duplicate tests on other fabrics are caused by real variations in the permeability of the fabric and not by unobserved errors of testing. If the fabric contains "pinholes," the variations in permeability of test pieces may be very large because it is unlikely that the small holes will be uniformly distributed and be uniform in size. One sample which showed

"pinholes" gave tests ranging from 14.5 to 43.5 liters. In general, duplicate tests on good fabrics show an agreement within approximately 1 liter in the range of 10 to 20 liters.

With the standard cell having an area of 250 cm^2 an interferometer giving the concentration of hydrogen to 0.01 per cent and a 1/20 cubic foot meter graduated in thousandths of a cubic foot and read to ten thousandths of a cubic foot, it is possible to keep the probable experimental errors below 5 per cent. Under routine testing conditions the accuracy may vary from 5 to 10 per cent. Greater variations than this in the uniformity of the fabric are commonly met with. The precision attained—that is, the agreement between successive tests on the same test piece—is somewhat greater than the absolute accuracy attained.

If the combustion method is used, about the same accuracy can be secured if the proper precautions to secure complete drying, combustion, and absorption are taken. Because of the length of time necessary for a test, check tests are not usually run, and furthermore it is necessary to obtain the average permeability over a period of several hours unless a very large cell is used.

10. SUMMARY

The various methods for determining the permeability of balloon fabrics to hydrogen are described in this paper; the precise and rapid method used at the Bureau of Standards is described in detail. The phenomenon of the passage of gases through rubber by solution in the rubber is discussed in relation to testing methods. Data are given to show the effect upon the apparent permeability of different experimental conditions such as temperature, pressure, humidity of the gas, duration of test, etc. A knowledge of the effect of these factors enables one to compare results of tests made under different conditions. It has been shown that methods which depend upon the loss of volume of hydrogen confined by a sample of the fabric give results which are about 70 per cent of those obtained by the standard method which measures the volume of hydrogen penetrating the fabric. This is due to the mutual permeability of the fabric to air and hydrogen.

The author is indebted to Messrs. Francis A. Smith, P. G. Ledig, S. F. Pickering, and I. L. Moore for their assistance in making the measurements reported.

WASHINGTON, March 16, 1918.

DEPARTMENT OF COMMERCE

TECHNOLOGIC PAPERS

OF THE

BUREAU OF STANDARDS

S. W. STRATTON, DIRECTOR

No. 114

A PORTABLE CUBIC-FOOT STANDARD FOR GAS

BY

M. H. STILLMAN, Assistant Physicist

Bureau of Standards

ISSUED JANUARY 28, 1919

PRICE, 5 CENTS

Sold only by the Superintendent of Documents, Government Printing Office
Washington, D. C.

WASHINGTON
GOVERNMENT PRINTING OFFICE
1919

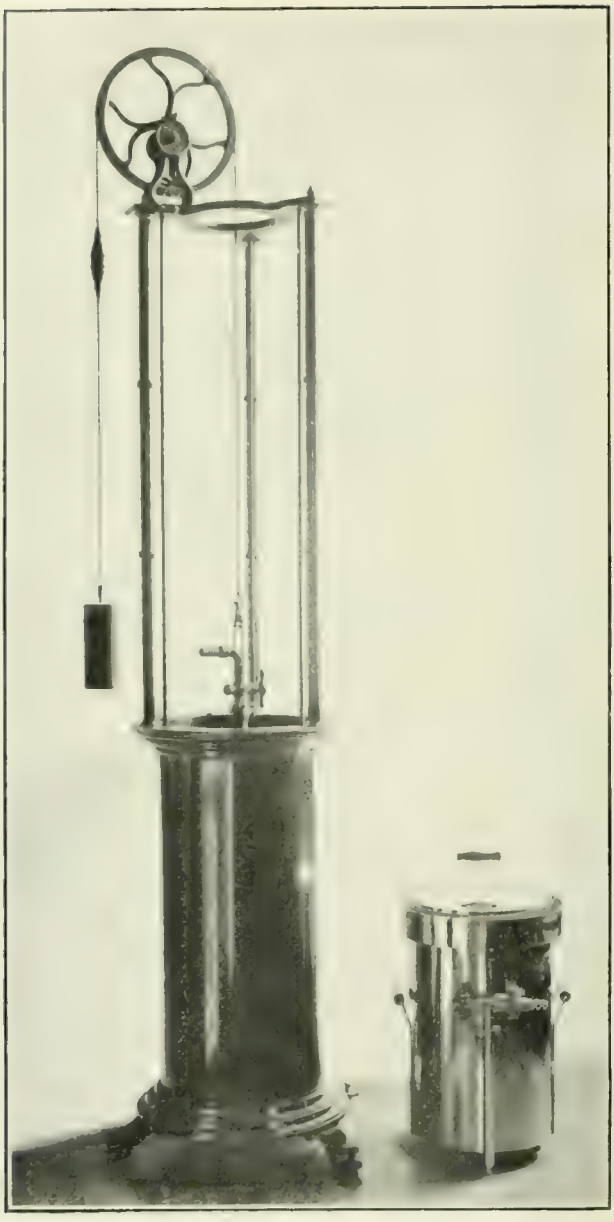


FIG. 1.—*Cubic foot apparatus*

The new cubic foot standard is shown at the right; at the left is shown one of the most compact types of cubic foot bottles commonly in use at the present time

A PORTABLE CUBIC-FOOT STANDARD FOR GAS

By M. H. Stillman

CONTENTS

	Page
I. Introduction	3
II. Description of standard	3
III. Operation	7
1. Calibration of provers	7
2. Use of new auxiliary meter-prover scale	11
3. Adjustment of cubic-foot standard	12
IV. Advantages of new standard	12
1. Portability	12
2. Accuracy	12
3. Convenience and rapidity	13

I. INTRODUCTION

The subject of this paper is a new type of volume standard for measuring gas which has been found to possess decided advantages as a cubic-foot standard over the apparatus of this class largely in use at the present time.[1]

While the portability, accuracy, and ease of operation of this device adapt it especially to that class of work in which an inspector is required to make tests of gas-meter testing equipment distributed over a comparatively large territory, it has also been successfully used for other purposes in which the delivery of a comparatively small volume of gas with a high degree of accuracy was required.

II. DESCRIPTION OF STANDARD

In Fig. 1, at the right, is shown a view of the new cubic-foot standard. It is about 25 inches high overall, and, in order to give an idea of its relative size, a cubic-foot bottle apparatus of

[1] A patent (No. 1 234 505) on this apparatus was granted to the author on July 24, 1917. This was dedicated to the public in the following words of the specification: "The invention herein described and claimed may be used by the Government of the United States, or by any of its officers or employees in the prosecution of work for the United States, or by any person in the United States, without payment of any royalty thereon."

Copies of this patent may be obtained for 5 cents each by making application to the Commissioner of Patents, Washington, D. C.

68057°—19

one of the more compact types at present in use is shown at the left. Fig. 2 is a vertical cross section through the axis of the apparatus, and Fig. 3 is a horizontal cross section at line *III–III* of Fig. 2.

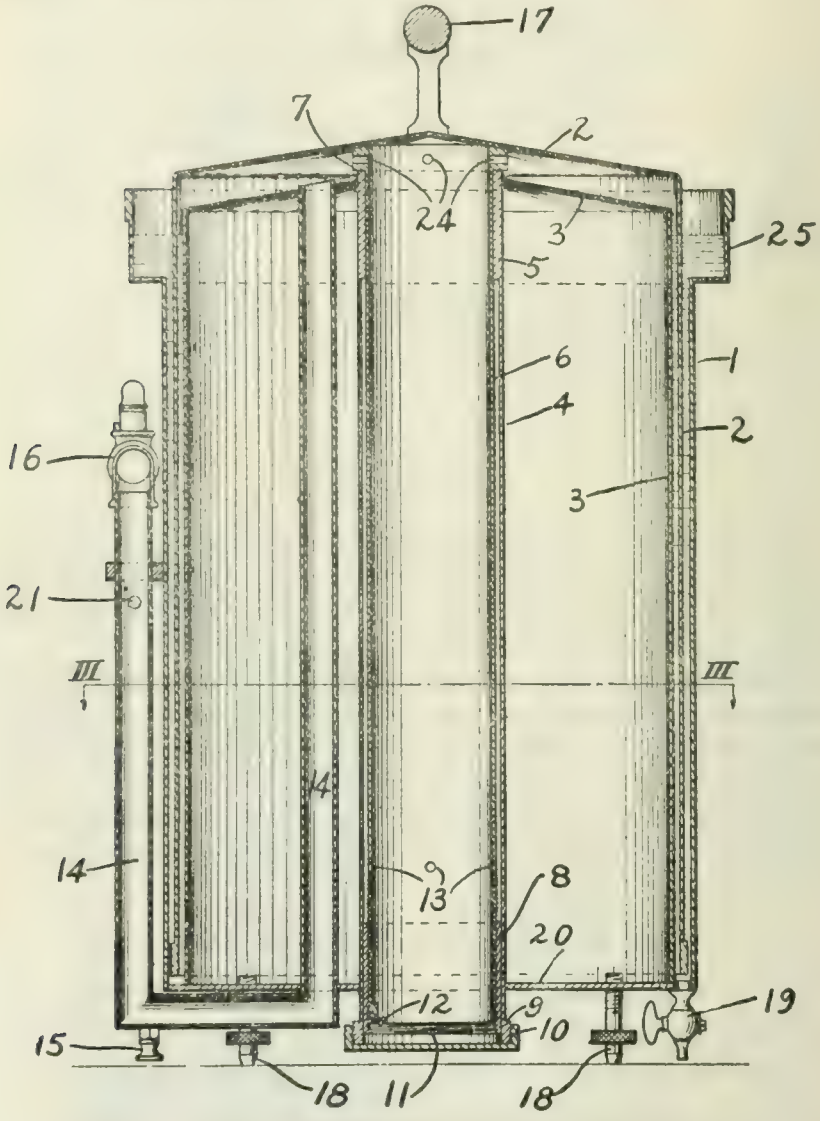


FIG. 2.—*Vertical cross section through cubic-foot bottle apparatus*

Referring to Figs. 2 and 3, *1* and *3* are coaxial cylinders, the annular space between them being closed at the bottom by the outer portion of the base plate *20*. This constitutes an annular tank in which the cylinder *2*, closed at the top as shown and

hereafter referred to as the bell, may be moved up and down, guided by the sliding mechanism at the axis of the apparatus. The inner cylinder *3* is also closed at the top and will be hereafter referred to as the core. The clearance space between the sides of the core and bell is made as small as is consistent with freedom of motion in order to minimize the changes in the volume of the sealing liquid within this space due to changes in the gas pressure within the bell. This results in rendering negligible the errors due to the small pressure changes that ordinarily occur. The

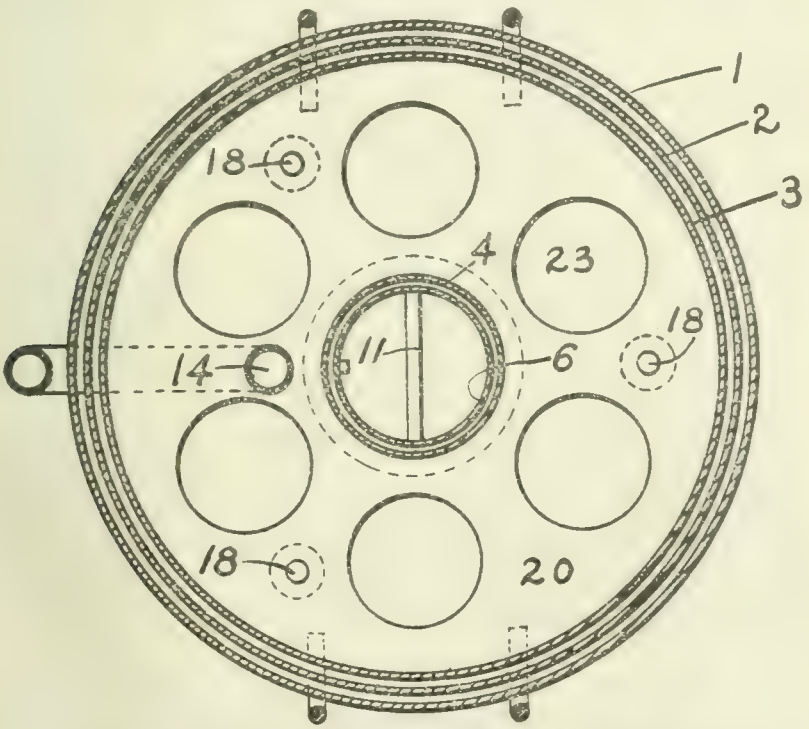


FIG. 3.—*Horizontal cross section through cubic-foot bottle apparatus*

diameter of the top of the outer cylinder *1* is enlarged to form a reservoir *25* of such capacity that the raising and lowering of the bell will not cause an excessive change in the height of the sealing liquid.

The guiding mechanism referred to above consists, primarily, of two telescoping tubes *4* and *6*, the inner tube *6* being securely fastened to the inside of the top of the bell, and consequently, as the bell is raised and lowered, sliding up and down within the stationary tube *4*, which is secured gas tight to the core and the base plate *20*. The interior of the upper end of the outside

guide tube is provided with a thickened portion or ring *5*, which is of such a thickness as to closely fit around the inside tube. The exterior of the lower end of the inside tube is provided with a screw thread to which is screwed, by means of a handle *11*, a ring or collar *8*, of such a thickness as to fit closely the inside surface of the outside tube. There is thus provided for the bell a guiding mechanism permitting very little lateral motion. It is evident that when the bell is raised by means of the handle *17* the upward motion will be limited by the collar *8* coming in contact with the shoulder formed by the collar *5*. It is also evident that the height to which the bell can be raised may be regulated by screwing the collar *8* up or down upon the inner tube. This collar when adjusted is fixed to the inside tube by means of the set screw *12*. The lowest position of the bell is established by the collar *7*, which is fastened to the bell and inside guide tube, coming in contact with the upper end of the outer stationary guide tube and its attached collar.

The wall of the lower end of the outside tube *4* is thickened and provided with an external screw thread to which is screwed the cap *10*, making therewith a gas-tight joint. In order to connect the interior of this guiding mechanism with the interior of the bell, two sets of holes *24* and *13* are provided.

To connect the interior of the bell with the free air or with any volume of gas, the tube *14* passes gas tight through the top of the core, down through the bottom *20*, and up to the valves *16*. The two valves *16*, which are clearly shown in Fig. 1, may advantageously be replaced by one three-way valve. To the opening *21* a manometer or other pressure-measuring device may be connected.

A stopcock *19* is connected to the bottom of the annular tank in order to provide a means of removing the sealing liquid which normally fills the tank up to approximately the height shown in Fig. 2.

The gas conductor *14* is provided with a drain cock *15* which is used to remove any sealing or other liquid which may accidentally enter the conductor.

Three screw feet are inserted into the bottom in order that the entire apparatus may easily be leveled.

A number of large holes *23*, Fig. 3, are placed in the bottom *20* in order to decrease the weight of the apparatus and to allow the air of the room to get within the core and thereby more quickly equalize the temperatures of the apparatus and the room.

While the instruments so far constructed have been either of brass or copper, aluminum could well have been utilized to a very large extent and the weight thereby materially reduced.

Only a little more than 1 gallon of sealing liquid is required by each instrument.

III. OPERATION

Briefly stated the method of operation is as follows: The annular tank is first filled with water or preferably an oil of small vapor pressure and small viscosity. The raising of the bell *2* to its upper limit by means of the handle *17* draws into the interior of the bell through the valve *16* and tube *14* a definite volume of gas. The magnitude of this volume depends for a given apparatus upon the distance the bell can be raised, which is the distance between the stops *5* and *8*. If the bell is then lowered to its initial position, it is obvious that the same definite volume of gas will be expelled.

The method of calibrating and adjusting the apparatus to deliver 1 cubic foot with the required accuracy will be described later; for the present it will be assumed that this adjustment has been made.

1. CALIBRATION OF PROVERS

The manner of using the apparatus to calibrate meter provers is so simple that those who have used the ordinary cubic-foot bottles would be able to use it successfully without further explanation. Use of the apparatus has, however, indicated certain details which should be observed if all the advantages inherent in this form of standard are to be realized. In order to aid in the discussion of these the following outline of procedure, which has been successfully used, is given:

1. Open one of the valves *16* to the air of the room and fill the annular tank with sealing liquid (preferably a proper oil) up to within about 1 inch of the top of the tank.

2. Connect one of the outlets *16* to the prover by means of a rubber tube.

3. Open all valves between prover and cubic-foot apparatus.

4. Test apparatus and connections for leaks.

5. Make necessary temperature adjustments.

6. Fill prover with air at normal prover pressure (usually 1½ inches of water) and adjust so that reading is zero.

7. Raise bell of standard from lowest position to highest position, thereby withdrawing 1 cubic foot of air from prover, read

prover, and record reading. (It will be necessary to keep the hand on the handle of the bell in order to maintain the bell in its highest position while taking this reading.)

8. Reintroduce into prover the cubic foot of air from standard by pushing bell down to lowest position and again make and record prover reading. Operations 7 and 8 effect two determinations of the interval 0–1 of the prover. Repeat these operations a sufficient number of times to determine the true volume of the interval 0–1 with the required accuracy.

9. With the bell of cubic-foot apparatus in lowest position allow 1 cubic foot of air to escape from the prover into the room and calibrate the next interval 1–2 of the prover in exactly the same manner as the interval 0–1 was calibrated.

10. Calibrate the other intervals in the same manner.

As is generally true when accurate comparisons of volume are to be made with air or gas as the medium of comparison, care must be taken to make proper allowance for difference in temperature and vapor content of the air in standard and prover. Generally the most satisfactory and practicable procedure is to have the temperature of all parts of the system involved in the comparison practically the same. If, as is advocated, a suitable oil is used as a sealing liquid in the standard, changes of temperature due to evaporation from the outside of the bell are practically zero. Also, because of the construction of the standard and the small amount of sealing liquid required by it, the temperature of the standard will be maintained at very nearly that of the room unless the room temperature varies rapidly. The advantages of this are evident. If, as advised under 6 of the above outline of procedure, the test is started with the bell of the prover full of air, the bell should remain in its highest position for some time before the test is started, allowing its surface to become dry and its interior to become saturated with water vapor (assuming that water is being used as a sealing liquid in the prover). By so doing the test may be conducted with practically a dry bell, the maximum wetted surface exposed to the air of the room at any time being that corresponding to 1 cubic foot of its volume, the average exposed wetted surface being only about one-half of this maximum.

In order to insure that the air in prover and cubic-foot apparatus is thoroughly mixed and saturated with water vapor, it is well to transfer a quantity of air back and forth between prover and

standard a number of times by raising and lowering the bell of the standard.

A good indication that the conditions are satisfactory for the commencement of the test is obtained by first noting the reading of the prover and then after interchanging air between standard and prover a few times noting the reading again. If the reading of the prover has not changed appreciably, it may be safely assumed that no serious leak exists and that the conditions of the air in prover and standard are, at least, temporarily stable.

It is very desirable to test the prover with the same air pressure in the bell and the same quantity of sealing liquid in the tank as obtain while the prover is being used to test meters. If this is not done, it is evident that the portion of the prover bell corresponding to a given interval on the scale while the prover is being tested will, in general, not be identical with the portion corresponding to the same interval while the prover is being used to test meters.[2] The cubic-foot standard under discussion is unusually well adapted to take advantage of this fact since its operation at ordinary prover pressures is the same as at atmospheric pressure.

In operating the standard, care must be taken not to raise or lower the bell too quickly. If it is raised too quickly, the air pressure inside the bell may be so much reduced that the sealing liquid will be drawn up over the core and into the gas conductor, or if at the commencement of the down stroke the bell is pushed down too rapidly, it will force the air out from the bottom of the bell. Of course, by increasing the size of the gas conductor or making it extend higher inside of the bell, the speed of the operation of the instrument may be greatly increased; but it has been found that even with the proportions shown in the drawing the apparatus may be easily and successfully operated considerably faster than the ordinary cubic-foot bottle.

There is given below a copy of the data sheet of the calibration of a 5-cubic-foot prover by the new standard. The prover bell was of painted sheet iron and was protected from the direct action of the air of the room by a stationary metal cover which was supported upon the rim of the prover tank. The sealing liquid used in both prover and cubic-foot standard was a thin oil which has been found very satisfactory.[3] The table shows the

[2] If the prover bell were of absolutely uniform diameter throughout that portion of its length exposed to the sealing liquid, this would be unimportant; but since variations do frequently occur in manufacture and through accidental denting, it is very desirable, as stated, to adopt a method of test that will avoid incorrect results due to such variations.

[3] The oil used as a sealing liquid is known as P. I. Meter Oil. It has the following physical characteristics: Flash point 65° C; fire point 95° C; viscosity 40 seconds at 100° F (Saybolt scale).

close agreement of successive determinations of the same interval that may be expected when proper precautions are taken. When water is used as a sealing liquid and the unsaturated air of the room is allowed to circulate freely about the bell of the prover, the results will be less accurate and the variations of successive determinations will be greater.

Data of Calibration of 5-Cubic-Foot Meter Prover

Interval on prover in cubic feet	Reading of prover scale		Volume of discharge indicated by prover	Temperature of cubic-foot standard	Temperature of prover	Temperature of air of room	Actual discharge for 1 cubic foot indicated by prover
	Before discharge	After discharge					
			Cubic feet	° C	° C	° C	Cubic feet
	0.000	0.998	0.998	21.5	21.5	22.0	
	.000	.997	997				
0–1 (1.30 p. m.)	.000	.9975	9975				
Mean			9975				1.002
	1.000	1.997	997				
	.999	1.996	997				
1–2	.999	1.996	.997				
Mean			.997				1.003
	2.000	3.000	1.000				
	2.000	3.000	1.000				
2–3	1.999	2.999	1.000				
Mean			1.000				1.000
	2.998	3.996	998				
	2.998	3.996	.998				
3–4	2.998	3.996	.998				
Mean			.998				1.002
	4.000	4.993	.993				
	4.000	4.993	.993				
4–5 (2.10 p. m.)	4.000	4.9925	.9925	21.9	21.9	22.1	
Mean			9928				1.007

Practically all of the methods of calibrating a prover at present in use, including that just described, involve the introduction into or withdrawal from the prover of a whole cubic foot of air, the only scale reading being that of the prover. As is well known, it is not always easy to read these scales accurately, say to one one-thousandth cubic foot, because of the length of the smallest interval (sometimes one-tenth cubic foot) and also because of the considerable width of the division marks. Some provers are provided with verniers by means of which the scales may be read to one one-thousandth of a cubic foot, but this is not common.

In order to avoid errors in reading due to the width of the graduations, the writer has found that the practice of reading either the top edges of all of the graduations of a given scale or the bottom edges of all the graduations is effective if the edges are straight and clearly defined.

2. USE OF NEW AUXILIARY METER-PROVER SCALE

For reading the prover scale with the accuracy desired in prover calibrations, the auxiliary scale described below has been used successfully for some time. While it was designed particularly for use in connection with the herein-described cubic-foot standard, it will be obvious that it is equally applicable where a prover is being calibrated by any form of cubic-foot standard.

As shown in Fig. 4, the auxiliary scale is simply a stationary scale *B* divided into divisions of such length as to correspond to one one-hundredth cubic foot on the prover scale *C*, and numbered as shown. This scale is then firmly mounted in front of and close to the prover scale. In reading the prover, the foot

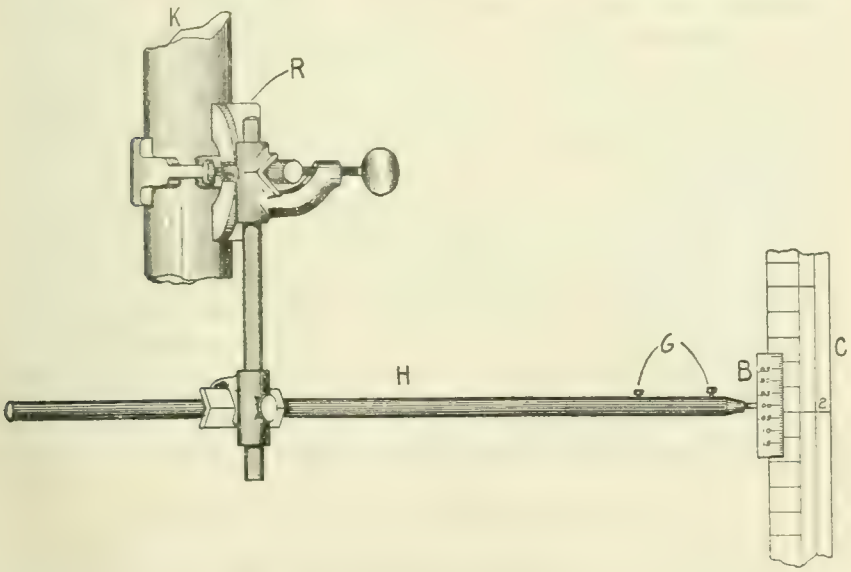


FIG. 4. – *Auxiliary meter-prover scale.*

division on the regular prover scale is considered as the index which moves under the stationary auxiliary scale. Thus the reading of the prover as illustrated in Fig. 4 is 2.026 cubic feet.

A convenient form of clamp permitting the easy adjustment of the auxiliary scale to the prover is also shown in Fig. 4 fixed to a column *K* of the prover. Four thumbscrews (not shown) on the right jaw *R* of the column clamp permit the adjustment of the clamp to the column whether this be of uniform or variable diameter. Inasmuch as provers of different diameters require scales of different sizes, provision is made for easily interchanging scales and firmly fastening them to the rod *H* by means of the thumbscrews *G*.

This scale makes it practicable to read the ordinary 5-cubic-foot prover to one one-thousandth cubic foot a degree of accuracy which is commensurate with the constancy of volume of gas in the prover bell which it is practicable to maintain for a short time under good laboratory conditions.

3. ADJUSTMENT OF CUBIC-FOOT STANDARD

The adjustment of the new cubic-foot standard to take in and deliver 1 cubic foot of air or other gas with a high degree of accuracy is easily made by making use of an accurately calibrated cubic-foot bottle.

The method consists of so adjusting the capacity of the new standard that upon discharging into it 1 cubic foot of air from the standardized cubic-foot bottle no change of pressure in the new standard takes place. As is evident from the description of the apparatus, the capacity of the standard is increased or decreased by screwing the adjustable guide and stop *8*, respectively, down or up.

The apparatus is well adapted to being officially adjusted and sealed. Sealing is accomplished either by sealing the adjustable lower guide *8* to the inner guide tube, thus preventing relative motion between the two, or by sealing the cap *10* to the outer guide tube in such a way that it can not be removed without breaking the seal. Moreover, with the bell in its lowest position, which it normally occupies except when a test is being made, it is almost completely protected from accidental injury.

IV. ADVANTAGES OF NEW STANDARD

Use of the cubic-foot standard described in this paper has shown it to be practicable and to possess to a marked degree the following advantages:

1. PORTABILITY

The apparatus is so compact and light that it can be carried about by one man. By providing it with a case it can safely be checked as baggage or shipped by express.

2. ACCURACY

While in itself this apparatus is not more accurate than the regulation cubic-foot bottle so commonly in use, yet it is believed that with the ordinary care that it is practicable to exercise in routine prover testing, the new apparatus will generally give more

accurate results than the former, because (1) it is practicable to use oil as a sealing liquid, since the small amount (little more than 1 gallon) can be easily carried with the apparatus or kept at each prover station. The oil does not evaporate from the surface of the bell to an appreciable extent and so introduce errors due to cooling and change of vapor pressure within the bell. (2) As explained above, it is very convenient to test the prover under the same conditions as regards pressure in the prover bell and quantity of sealing liquid in the tank as obtain during routine meter testing. (3) The transfer of air between prover and standard is accomplished very quickly, thus reducing variations in temperature and vapor pressure within the prover and standard during the test of a given interval.

3. CONVENIENCE AND RAPIDITY

Setting up the new standard for a test consists simply in removing it from the case and pouring the necessary quantity of oil (or other sealing liquid) into the tank. This requires only a few moments. After completing the test of one prover the standard may be easily moved to the next by one man without removing the sealing liquid. The operation of calibration itself is rapid; considerably faster than is practicable with the ordinary cubic-foot bottle.

WASHINGTON, January 1, 1918.

DEPARTMENT OF COMMERCE

BUREAU OF STANDARDS

S. W. STRATTON, Director

TECHNOLOGIC PAPERS OF THE BUREAU OF STANDARDS NO. 115

[Issued October 30, 1918]

NEW BAUMÉ SCALE FOR SUGAR SOLUTIONS

By Frederick J. Bates, Chemist, and H. W. Bearce, Associate Physicist

In laboratory and refinery measurements on sugar solutions it is customary to determine the percentage of sugar either by means of a picnometer or a hydrometer. In case the picnometer is used it is customary to obtain the specific gravity at some specified temperature in terms of water at the same temperature as unity, and then by means of a conversion table to find the corresponding percentage of sugar. In case the hydrometer is used it is customary either to read the percentage of sugar direct or to read the degrees Baumé and then, by an appropriate conversion table, to determine the corresponding percentage of sugar. In certain cases the Baumé degrees are used without reference to any other scale, but in general a definite relation between degrees Baumé and degrees Brix or specific gravity must be agreed upon before the Baumé readings are of any great value. This relation is arrived at by determining experimentally the specific gravity corresponding to various known percentages of sugar and then calculating the degrees Baumé corresponding to these specific gravities by assuming a constant multiplier or "modulus" on which the Baumé scale is based. Having chosen the modulus, the working out of the table relating specific gravity and degrees Baumé is purely arithmetical, the degrees Baumé corresponding to a series of specific gravities being calculated by means of the equation

$$d = m - \frac{m}{s},$$

in which d = degrees Baumé, m = modulus, and s = specific gravity. The specific gravities must all be reduced to the same temperature and it is desirable also that they be expressed in terms of water at the same temperature, in order that zero per cent sugar may have a specific gravity of unity. This will give also a value of zero degrees Baumé corresponding to zero per cent sugar, or zero degrees Brix.[1]

Many different Baumé scales have been proposed and used in the past, and at the present time there are still in use in the United States three different scales for liquids heavier than water. One of these, the American standard Baumé scale, is based on the modulus 145 and on the temperature 60° F, and is extensively used in the acid industry and in other similar lines with the exception of the sugar industry. (See Circular No. 59, Bureau of Standards.) The other two scales, the "old" or "Holland" scale and the "new" or "Gerlach" scale, are used in sugar work. The Gerlach scale, with the modulus 146.78 and the temperature 17.5° C seems to be most often employed. In the "Methods of analysis" of the Association of Official Agricultural Chemists both tables are given, the more extensive of the two being the "old" or "Holland" scale based on the modulus 144.

The new table lies between the so-called "old" and "new" tables, being in almost perfect agreement with the "old" up to 25 per cent sugar and from that point on about 0.1° Baumé higher than the "old." A brief comparison between the three tables is given in Table 1.

[1] Hydrometers are not certified by the Bureau of Standards on the basis of the original "Brix" or "Balling" scale. Both "Brix" and "Balling" are interpreted by the Bureau as meaning percentage of pure sucrose of weight.

TABLE 1.—Comparison of Baumé Scales

Per cent sucrose or degrees Brix	Corresponding degrees Baumé		
	"New" scale (modulus 146.78)	"Old" scale (modulus 144)	B. S. scale (modulus 145)
0	0.0	0.0	0.00
5	2.8	2.8	2.79
10	5.7	5.6	5.57
15	8.5	8.3	8.34
20	11.3	11.1	11.10
25	14.1	13.8	13.84
30	16.8	16.5	16.57
35	19.6	19.2	19.28
40	22.3	21.9	21.97
45	25.0	24.6	24.63
50	27.7	27.2	27.28
55	30.4	29.8	29.90
60	33.0	32.4	32.49
65	35.6	34.9	35.04
70	38.1	37.4	37.56
75	40.6	39.9	40.03
80	43.1	42.3	42.47
85	45.5	44.7	44.86
90	47.9	47.0	47.20
95	50.3	49.3	49.49
100	.....	.....	51.73

The Bureau of Standards has always held[1] that the relation between specific gravity and degrees Baumé, for liquids heavier than water, should be that based on the modulus 145. This modulus is now in universal use in the United States except in the sugar industry. It has been adopted by the Manufacturing Chemists Association, by the Bureau of Standards, and by all American manufacturers of Baumé hydrometers.

The American standard Baumé scale, as heretofore used, is based on specific gravities at 60°/60° F instead of 20°/20° C, but this change should not be considered serious, since the same numerical relation between specific gravity and degrees Baumé will still hold; that is, the degrees Baumé corresponding to a given specific gravity will still be the same as before. The only difference is that the specific gravity 20°/20° C corresponding to any given percentage of sugar is slightly different from what it would be at 60°/60° F.

The relation between specific gravity and degrees Baumé is as follows:

$$\text{Degrees Baumé} = 145 - \frac{145}{\text{Specific gravity}}$$

[1] See Bureau of Standards Circulars Nos. 10, 19, and 59.

The Baumé scale here presented has three features which should commend it for use in sugar work.

1. It is based upon the specific gravity values of Plato [2] which are considered the most reliable of any available.

2. It is based on 20° C, the most convenient and widely accepted temperature for sugar work.

3. It is based on the modulus 145, which has already been adopted by the Manufacturing Chemists Association of the United States, by the Bureau of Standards, and by all American manufacturers of hydrometers.

TABLE 2.—Degrees Brix, Specific Gravity, and Degrees Baumé of Sugar Solutions

Degrees Brix or per cent sucrose by weight	Specific gravity at 20°/4° C	Specific gravity at 20° 20° C	Degrees Baumé (modulus 145)	Degrees Brix or per cent sucrose by weight	Specific gravity at 20°/4° C	Specific gravity at 20°,20° C	Degrees Baumé (modulus 145)
[illegible]	[illegible]	1.00000	0.00	4.5	1.01586	1.01766	2.52
[illegible]	[illegible]	1.00039	0.06	4.6	1.01626	1.01806	2.57
[illegible]	[illegible]	1.00078	0.11	4.7	1.01666	1.01846	2.63
0.3	[illegible]	1.00117	0.17	4.8	1.01706	[illegible]	2.68
0.4	[illegible]	1.00155	0.22	4.9	1.01746	1.01926	2.74
0.5	[illegible]	1.00194	0.28	5.0	1.01785	1.01965	2.79
0.6	1.00056	1.00233	0.34	5.1	1.01825	1.02005	2.85
0.7	1.00095	1.00272	0.39	5.2	1.01865	1.02045	2.91
0.8	1.00134	1.00311	0.45	5.3	1.01905	1.02085	2.96
0.9	1.00173	1.00350	0.51	5.4	1.01945	1.02125	3.02
1.0	1.00212	1.00389	0.56	5.5	1.01985	1.02165	3.07
1.1	1.00251	1.00428	0.62	5.6	1.02025	1.02206	3.13
1.2	1.00290	1.00467	0.67	5.7	[illegible]	1.02246	3.18
1.3	1.00329	[illegible]	0.73	5.8	1.02105	1.02286	3.24
1.4	1.00368	1.00545	0.79	5.9	1.02145	1.02326	3.30
1.5	[illegible]	1.00584	0.84	6.0	1.02186	1.02366	3.35
1.6	1.00445	1.00623	0.90	6.1	[illegible]	1.02407	3.41
1.7	1.00484	1.00662	0.95	6.2	1.02266	1.02447	3.46
1.8	1.00523	1.00701	1.01	6.3	[illegible]	1.02487	3.52
1.9	1.00562	1.00740	1.07	6.4	1.02346	1.02527	3.57
[illegible]	1.00602	1.00779	1.12	6.5	1.02387	1.02568	3.63
2.1	[illegible]	1.00818	1.18	6.6	1.02427	1.02608	3.69
2.2	1.00680	1.00858	1.23	6.7	1.02467	1.02648	3.74
2.3	1.00719	1.00897	1.29	6.8	1.02508	[illegible]	3.80
[illegible]	[illegible]	[illegible]	1.34	6.9	1.02548	1.02729	3.85
2.5	1.00797	[illegible]	1.40	7.0	1.02588	1.02770	3.91
2.6	1.00836	1.01015	1.46	7.1	[illegible]	1.02810	3.96
2.7	1.00876	1.01054	1.51	7.2	[illegible]	1.02851	4.02
2.8	1.00915	[illegible]	1.57	7.3	[illegible]	1.02892	4.08
2.9	1.00954	1.01133	1.62	7.4	1.02750	1.02932	4.13
3.0	1.00993	1.01172	1.68	7.5	[illegible]	1.02973	4.19
3.1	1.01033	1.01211	1.74	7.6	[illegible]	1.03013	4.24
3.2	1.01072	1.01251	1.79	7.7	1.02872	1.03054	4.30
3.3	1.01112	[illegible]	1.85	7.8	1.02913	1.03095	4.35
3.4	1.01151	1.01330	1.90	7.9	1.02954	1.03136	4.41
3.5	1.01190	1.01369	1.96	8.0	1.02994	1.03176	4.46
3.6	1.01230	1.01409	2.02	8.1	1.03035	1.03217	4.52
3.7	1.01269	1.01448	2.07	8.2	1.03076	1.03258	4.58
3.8	[illegible]	1.01488	2.13	8.3	[illegible]	[illegible]	4.63
3.9	1.01348	1.01528	2.18	8.4	1.03157	1.03340	4.69
4.0	1.01388	1.01567	2.24	8.5	1.03198	1.03381	4.74
4.1	1.01428	1.01607	2.29	8.6	[illegible]	[illegible]	4.80
4.2	1.01467	1.01647	2.35	8.7	1.03280	1.03463	4.85
4.3	1.01507	[illegible]	2.40	8.8	1.03321	1.03504	4.91
4.4	1.01547	1.01726	2.46	8.9	1.03362	1.03545	4.96

[2] Dr. F. Plato, Wiss. Abh. Der Kaiserlichen Normal-Eichungs-Kommission, 2, p. 153; 1900.

TABLE 2—Continued

Degrees Brix or per cent sucrose by weight	Specific gravity at 20°/4° C	Specific gravity at 20°/20° C	Degrees Baumé (modulus 145)	Degrees Brix or per cent sucrose by weight	Specific gravity at 20°/4° C	Specific gravity at 20°/20° C	Degrees Baumé (modulus 145)
9.0	1.03403	1.03586	5.02	15.5	1.06131	1.06319	8.62
9.1	1.03444	1.03627	5.07	15.6	1.06174	1.06362	8.67
9.2	1.03485	1.03668	5.13	15.7	1.06217	1.06405	8.73
9.3	1.03526	1.03709	5.19	15.8	1.06260	1.06448	8.78
9.4	1.03567	1.03750	5.24	15.9	1.06303	1.06491	8.84
9.5	1.03608	1.03792	5.30	16.0	1.06346	1.06534	8.89
9.6	1.03649	1.03833	5.35	16.1	1.06389	1.06577	8.95
9.7	1.03691	1.03874	5 41	16.2	1.06432	1.06621	9.00
9.8	1.03732	1.03915	5.46	16.3	1.06476	1.06664	9.06
9.9	1.03773	1.03957	5.52	16.4	1.06519	1.06707	9.11
10.0	1.03814	1.03998	5.57	16.5	1.06562	1.06751	9.17
10.1	1.03856	1.04039	5.63	16.6	1.06605	1.06794	9.22
10.2	1.03897	1.04081	5.68	16.7	1.06649	1.06837	9.28
10.3	1.03938	1.04122	5.74	16.8	1.06692	1.06881	9.33
10.4	1.03980	1.04164	5.80	16.9	1.06736	1.06924	9.39
10.5	1.04021	1.04205	5.85	17.0	1.06779	1.06968	9.45
10.6	1.04063	1.04247	5.91	17.1	1.06822	1.07011	9.50
10.7	1.04104	1.04288	5.96	17.2	1.06866	1.07055	9.56
10.8	1.04146	1.04330	6.02	17.3	1.06909	1.07098	9.61
10.9	1.04187	1.04371	6.07	17.4	1.06953	1.07142	9.67
11.0	1.04229	1.04413	6.13	17.5	1.06996	1.07186	9.72
11.1	1.04270	1.04455	6.18	17.6	1.07040	1.07229	9.78
11.2	1.04312	1.04497	6.24	17.7	1.07084	1.07273	9.83
11.3	1.04354	1.04538	6.30	17.8	1.07127	1.07317	9.89
11.4	1.04395	1.04580	6.35	17.9	1.07171	1.07361	9.94
11.5	1.04437	1.04622	6.41	18.0	1.07215	1.07404	10.00
11.6	1.04479	1.04664	6.46	18.1	1.07258	1.07448	10.05
11.7	1.04521	1.04706	6.52	18.2	1.07302	1.07492	10.11
11.8	1.04562	1.04747	6.57	18.3	1.07346	1.07536	10.16
11.9	1.04604	1.04789	6.63	18.4	1.07390	1.07580	10.22
12.0	1.04646	1.04831	6.68	18.5	1.07434	1.07624	10.27
12.1	1.04688	1.04873	6.74	18.6	1.07478	1.07668	10.33
12.2	1.04730	1.04915	6.79	18.7	1.07522	1.07712	10.38
12.3	1.04772	1.04957	6.85	18 8	1.07566	1.07756	10.44
12.4	1.04814	1.04999	6.90	18.9	1.07610	1.07800	10.49
12.5	1.04856	1.05041	6.96	19.0	1.07654	1.07844	10.55
12.6	1.04898	1.05084	7.02	19.1	1.07698	1.07888	10.60
12.7	1.04940	1.05126	7.07	19.2	1.07742	1.07932	10.66
12.8	1.04982	1.05168	7.13	19.3	1.07786	1.07977	10.71
12.9	1.05024	1.05210	7.18	19.4	1.07830	1.08021	10.77
13.0	1.05066	1.05252	7.24	19.5	1.07874	1.08065	10.82
13.1	1.05109	1.05295	7.29	19.6	1.07919	1.08110	10.88
13.2	1.05151	1.05337	7.35	19.7	1.07963	1.08154	10.93
13.3	1.05193	1.05379	7.40	19.8	1.08007	1.08198	10.99
13.4	1.05236	1.05422	7.46	19.9	1.08052	1.08243	11.04
13.5	1.05278	1.05464	7.51	20.0	1.08096	1.08287	11.10
13.6	1.05320	1.05506	7.57	20.1	1.08140	1.08332	11.15
13.7	1.05363	1.05549	7.62	20.2	1.08185	1.08376	11.21
13.8	1.05405	1.05591	7.68	20.3	1.08229	1.08421	11.26
13.9	1.05448	1.05634	7.73	20.4	1.08274	1.08465	11.32
14.0	1.05490	1.05677	7.79	20.5	1.08318	1.08510	11.37
14.1	1.05532	1.05719	7.84	20.6	1.08363	1.08554	11.43
14.2	1.05575	1.05762	7.90	20.7	1.08407	1.08599	11.48
14.3	1.05618	1.05804	7.96	20.8	1.08452	1.08644	11.54
14.4	1.05660	1.05847	8.01	20.9	1.08497	1.08689	11.59
14.5	1.05703	1.05890	8.07	21.0	1.08541	1.08733	11.65
14.6	1.05746	1.05933	8.12	21.1	1.08586	1.08778	11.70
14.7	1.05788	1.05975	8.18	21.2	1.08631	1.08823	11.76
14.8	1.05831	1.06018	8.23	21.3	1.08676	1.08868	11.81
14.9	1.05874	1.06061	8.29	21.4	1.08720	1.08913	11.87
15.0	1.05916	1.06104	8.34	21.5	1.08765	1.08958	11.92
15.1	1.05959	1.06147	8.40	21.6	1.08810	1.09003	11.98
15.2	1.06002	1.06190	8.45	21.7	1.08855	1.09048	12.03
15.3	1.06045	1.06233	8.51	21.8	1.08900	1.09093	12.09
15.4	1.06088	1.06276	8.56	21.9	1.08945	1.09138	12.14

TABLE 2 - Continued

Degrees Brix or per cent sucrose by weight	Specific gravity at 20°/4° C	Specific gravity at 20°/20° C	Degrees Baumé (modulus 145)	Degrees Brix or per cent sucrose by weight	Specific gravity at 20°/4° C	Specific gravity at 20°/20° C	Degrees Baumé (modulus 145)
22.0	1.08990	1.09183	12.20	28.5	1.11987	1.12185	15.75
22.1	1.09035	1.09228	12.25	28.6	1.12034	1.12232	15.80
22.2	1.09080	1.09273	12.31	28.7	1.12081	1.12280	15.86
22.3	1.09125	1.09318	12.36	28.8	1.12128	1.12327	15.91
22.4	1.09170	1.09364	12.42	28.9	1.12176	1.12374	15.97
22.5	1.09216	1.09409	12.47	29.0	1.12223	1.12422	16.02
22.6	1.09261	1.09454	12.52	29.1	1.12270	1.12469	16.08
22.7	1.09306	1.09499	12.58	29.2	1.12318	1.12517	16.13
22.8	1.09351	1.09545	12.63	29.3	1.12365	1.12564	16.18
22.9	1.09397	1.09590	12.69	29.4	1.12413	1.12612	16.24
23.0	1.09442	1.09636	12.74	29.5	1.12460	1.12659	16.29
23.1	1.09487	1.09681	12.80	29.6	1.12508	1.12707	16.35
23.2	1.09533	1.09727	12.85	29.7	1.12556	1.12755	16.40
23.3	1.09578	1.09772	12.91	29.8	1.12603	1.12802	16.46
23.4	1.09624	1.09818	12.96	29.9	1.12651	1.12850	16.51
23.5	1.09669	1.09863	13.02	30.0	1.12698	1.12898	16.57
23.6	1.09715	1.09909	13.07	30.1	1.12746	1.12946	16.62
23.7	1.09760	1.09954	13.13	30.2	1.12794	1.12993	16.67
23.8	1.09806	1.10000	13.18	30.3	1.12842	1.13041	16.73
23.9	1.09851	1.10046	13.24	30.4	1.12890	1.13089	16.78
24.0	1.09897	1.10092	13.29	30.5	1.12937	1.13137	16.84
24.1	1.09943	1.10137	13.35	30.6	1.12985	1.13185	16.89
24.2	1.09989	1.10183	13.40	30.7	1.13033	1.13233	16.95
24.3	1.10034	1.10229	13.46	30.8	1.13081	1.13281	17.00
24.4	1.10080	1.10275	13.51	30.9	1.13129	1.13329	17.05
24.5	1.10126	1.10321	13.57	31.0	1.13177	1.13378	17.11
24.6	1.10172	1.10367	13.62	31.1	1.13225	1.13426	17.16
24.7	1.10218	1.10413	13.67	31.2	1.13274	1.13474	17.22
24.8	1.10264	1.10459	13.73	31.3	1.13322	1.13522	17.27
24.9	1.10310	1.10505	13.78	31.4	1.13370	1.13570	17.33
25.0	1.10356	1.10551	13.84	31.5	1.13418	1.13619	17.38
25.1	1.10402	1.10597	13.89	31.6	1.13466	1.13667	17.43
25.2	1.10448	1.10643	13.95	31.7	1.13515	1.13715	17.49
25.3	1.10494	1.10689	14.00	31.8	1.13563	1.13764	17.54
25.4	1.10540	1.10736	14.06	31.9	1.13611	1.13812	17.60
25.5	1.10586	1.10782	14.11	32.0	1.13660	1.13861	17.65
25.6	1.10632	1.10828	14.17	32.1	1.13708	1.13909	17.70
25.7	1.10679	1.10874	14.22	32.2	1.13756	1.13958	17.76
25.8	1.10725	1.10921	14.28	32.3	1.13805	1.14006	17.81
25.9	1.10771	1.10967	14.33	32.4	1.13853	1.14055	17.87
26.0	1.10818	1.11014	14.39	32.5	1.13902	1.14103	17.92
26.1	1.10864	1.11060	14.44	32.6	1.13951	1.14152	17.98
26.2	1.10910	1.11106	14.49	32.7	1.13999	1.14201	18.03
26.3	1.10957	1.11153	14.55	32.8	1.14048	1.14250	18.08
26.4	1.11003	1.11200	14.60	32.9	1.14097	1.14298	18.14
26.5	1.11050	1.11246	14.66	33.0	1.14145	1.14347	18.19
26.6	1.11096	1.11293	14.71	33.1	1.14194	1.14396	18.25
26.7	1.11143	1.11339	14.77	33.2	1.14243	1.14445	18.30
26.8	1.11190	1.11386	14.82	33.3	1.14292	1.14494	18.36
26.9	1.11236	1.11433	14.88	33.4	1.14340	1.14543	18.41
27.0	1.11283	1.11480	14.93	33.5	1.14389	1.14592	18.46
27.1	1.11330	1.11526	14.99	33.6	1.14438	1.14641	18.52
27.2	1.11376	1.11573	15.04	33.7	1.14487	1.14690	18.57
27.3	1.11423	1.11620	15.09	33.8	1.14536	1.14739	18.63
27.4	1.11470	1.11667	15.15	33.9	1.14585	1.14788	18.68
27.5	1.11517	1.11714	15.20	34.0	1.14634	1.14837	18.73
27.6	1.11564	1.11761	15.26	34.1	1.14684	1.14886	18.79
27.7	1.11610	1.11808	15.31	34.2	1.14733	1.14936	18.84
27.8	1.11657	1.11855	15.37	34.3	1.14782	1.14985	18.90
27.9	1.11704	1.11902	15.42	34.4	1.14831	1.15034	18.95
28.0	1.11751	1.11949	15.48	34.5	1.14880	1.15084	19.00
28.1	1.11798	1.11996	15.53	34.6	1.14930	1.15133	19.06
28.2	1.11845	1.12043	15.59	34.7	1.14979	1.15183	19.11
28.3	1.11892	1.12090	15.64	34.8	1.15029	1.15232	19.17
28.4	1.11940	1.12138	15.69	34.9	1.15078	1.15282	19.22

TABLE 2.—Continued

Degrees Brix or per cent sucrose by weight	Specific gravity at 20°/4° C	Specific gravity at 20°/20° C	Degrees Baumé (modulus 145)	Degrees Brix or per cent sucrose by weight	Specific gravity at 20°/4° C	Specific gravity at 20°/20° C	Degrees Baumé (modulus 145)
35.0	1.15128	1.15331	19.28	41.5	1.18418	1.18627	22.77
35.1	1.15177	1.15381	19.33	41.6	1.18470	1.18679	22.82
35.2	1.15226	1.15430	19.38	41.7	1.18522	1.18731	22.88
35.3	1.15276	1.15480	19.44	41.8	1.18573	1.18783	22.93
35.4	1.15326	1.15530	19.49	41.9	1.18625	1.18835	22.98
35.5	1.15375	1.15579	19.55	42.0	1.18677	1.18887	23.04
35.6	1.15425	1.15629	19.60	42.1	1.18729	1.18939	23.09
35.7	1.15475	1.15679	19.65	42.2	1.18781	1.18992	23.14
35.8	1.15524	1.15729	19.71	42.3	1.18834	1.19044	23.20
35.9	1.15574	1.15778	19.76	42.4	1.18886	1.19096	23.25
36.0	1.15624	1.15828	19.81	42.5	1.18938	1.19148	23.30
36.1	1.15674	1.15878	19.87	42.6	1.18990	1.19201	23.36
36.2	1.15724	1.15928	19.92	42.7	1.19042	1.19253	23.41
36.3	1.15773	1.15978	19.98	42.8	1.19095	1.19305	23.46
36.4	1.15823	1.16028	20.03	42.9	1.19147	1.19358	23.52
36.5	1.15873	1.16078	20.08	43.0	1.19199	1.19410	23.57
36.6	1.15923	1.16128	20.14	43.1	1.19252	1.19463	23.62
36.7	1.15973	1.16178	20.19	43.2	1.19304	1.19515	23.68
36.8	1.16023	1.16228	20.25	43.3	1.19356	1.19568	23.73
36.9	1.16073	1.16279	20.30	43.4	1.19409	1.19620	23.78
37.0	1.16124	1.16329	20.35	43.5	1.19462	1.19673	23.84
37.1	1.16174	1.16379	20.41	43.6	1.19514	1.19726	23.89
37.2	1.16224	1.16430	20.46	43.7	1.19567	1.19778	23.94
37.3	1.16274	1.16480	20.52	43.8	1.19619	1.19831	24.00
37.4	1.16324	1.16530	20.57	43.9	1.19672	1.19884	24.05
37.5	1.16375	1.16581	20.62	44.0	1.19725	1.19936	24.10
37.6	1.16425	1.16631	20.68	44.1	1.19778	1.19989	24.16
37.7	1.16476	1.16682	20.73	44.2	1.19830	1.20042	24.21
37.8	1.16526	1.16732	20.78	44.3	1.19883	1.20095	24.26
37.9	1.16576	1.16783	20.84	44.4	1.19936	1.20148	24.32
38.0	1.16627	1.16833	20.89	44.5	1.19989	1.20201	24.37
38.1	1.16678	1.16884	20.94	44.6	1.20042	1.20254	24.42
38.2	1.16728	1.16934	21.00	44.7	1.20095	1.20307	24.48
38.3	1.16779	1.16985	21.05	44.8	1.20148	1.20360	24.53
38.4	1.16829	1.17036	21.11	44.9	1.20201	1.20414	24.58
38.5	1.16880	1.17087	21.16	45.0	1.20254	1.20467	24.63
38.6	1.16931	1.17138	21.21	45.1	1.20307	1.20520	24.69
38.7	1.16982	1.17188	21.27	45.2	1.20360	1.20573	24.74
38.8	1.17032	1.17239	21.32	45.3	1.20414	1.20627	24.79
38.9	1.17083	1.17290	21.38	45.4	1.20467	1.20680	24.85
39.0	1.17134	1.17341	21.43	45.5	1.20520	1.20733	24.90
39.1	1.17185	1.17392	21.48	45.6	1.20573	1.20787	24.95
39.2	1.17236	1.17443	21.54	45.7	1.20627	1.20840	25.01
39.3	1.17287	1.17494	21.59	45.8	1.20680	1.20894	25.06
39.4	1.17338	1.17545	21.64	45.9	1.20734	1.20947	25.11
39.5	1.17389	1.17596	21.70	46.0	1.20787	1.21001	25.17
39.6	1.17440	1.17648	21.75	46.1	1.20840	1.21054	25.22
39.7	1.17491	1.17699	21.80	46.2	1.20894	1.21108	25.27
39.8	1.17542	1.17750	21.86	46.3	1.20948	1.21162	25.32
39.9	1.17594	1.17802	21.91	46.4	1.21001	1.21215	25.38
40.0	1.17645	1.17853	21.97	46.5	1.21055	1.21269	25.43
40.1	1.17696	1.17904	22.02	46.6	1.21109	1.21323	25.48
40.2	1.17747	1.17956	22.07	46.7	1.21162	1.21377	25.54
40.3	1.17799	1.18007	22.13	46.8	1.21216	1.21431	25.59
40.4	1.17850	1.18058	22.18	46.9	1.21270	1.21484	25.64
40.5	1.17901	1.18110	22.23	47.0	1.21324	1.21538	25.70
40.6	1.17953	1.18162	22.29	47.1	1.21378	1.21592	25.75
40.7	1.18004	1.18213	22.34	47.2	1.21432	1.21646	25.80
40.8	1.18056	1.18265	22.39	47.3	1.21486	1.21700	25.86
40.9	1.18108	1.18316	22.45	47.4	1.21540	1.21755	25.91
41.0	1.18159	1.18368	22.50	47.5	1.21594	1.21809	25.96
41.1	1.18211	1.18420	22.55	47.6	1.21648	1.21863	26.01
41.2	1.18262	1.18472	22.61	47.7	1.21702	1.21917	26.07
41.3	1.18314	1.18524	22.66	47.8	1.21756	1.21971	26.12
41.4	1.18366	1.18575	22.72	47.9	1.21810	1.22026	26.17

TABLE 2—Continued

Degrees Brix or per cent sucrose by weight	Specific gravity at 20° 4° C	Specific gravity at 20° 20° C	Degrees Baumé (modulus 145)	Degrees Brix or per cent sucrose by weight	Specific gravity at 20°/4° C	Specific gravity at 20°/20° C	Degrees Baumé (modulus 145)
48.0	1.21864	1.22080	26.23	54.5	1.25470	1.25692	29.64
48.1	1.21918	1.22134	26.28	54.6	1.25526	1.25748	29.69
48.2	1.21973	1.22189	26.33	54.7	1.25583	1.25805	29.74
48.3	1.22027	1.22243	26.38	54.8	1.25640	1.25862	29.80
48.4	1.22082	1.22298	26.44	54.9	1.25697	1.25919	29.85
48.5	1.22136	1.22352	26.49	55.0	1.25754	1.25976	29.90
48.6	1.22190	1.22406	26.54	55.1	1.25810	1.26033	29.95
48.7	1.22245	1.22461	26.59	55.2	1.25867	1.26090	30.00
48.8	1.22300	1.22516	26.65	55.3	1.25924	1.26147	30.06
48.9	1.22354	1.22570	26.70	55.4	1.25982	1.26204	30.11
49.0	1.22409	1.22625	26.75	55.5	1.26039	1.26261	30.16
49.1	1.22463	1.22680	26.81	55.6	1.26096	1.26319	30.21
49.2	1.22518	1.22735	26.86	55.7	1.26153	1.26376	30.26
49.3	1.22573	1.22789	26.91	55.8	1.26210	1.26433	30.32
49.4	1.22627	1.22844	26.96	55.9	1.26267	1.26490	30.37
49.5	1.22682	1.22899	27.02	56.0	1.26324	1.26548	30.42
49.6	1.22737	1.22954	27.07	56.1	1.26382	1.26605	30.47
49.7	1.22792	1.23009	27.12	56.2	1.26439	1.26663	30.52
49.8	1.22847	1.23064	27.18	56.3	1.26496	1.26720	30.57
49.9	1.22902	1.23119	27.23	56.4	1.26554	1.26778	30.63
50.0	1.22957	1.23174	27.28	56.5	1.26611	1.26835	30.68
50.1	1.23012	1.23229	27.33	56.6	1.26669	1.26893	30.73
50.2	1.23067	1.23284	27.39	56.7	1.26726	1.26950	30.78
50.3	1.23122	1.23340	27.44	56.8	1.26784	1.27008	30.83
50.4	1.23177	1.23395	27.49	56.9	1.26841	1.27066	30.89
50.5	1.23232	1.23450	27.54	57.0	1.26899	1.27123	30.94
50.6	1.23287	1.23506	27.60	57.1	1.26956	1.27181	30.99
50.7	1.23343	1.23561	27.65	57.2	1.27014	1.27239	31.04
50.8	1.23398	1.23616	27.70	57.3	1.27072	1.27297	31.09
50.9	1.23453	1.23672	27.75	57.4	1.27130	1.27355	31.15
51.0	1.23508	1.23727	27.81	57.5	1.27188	1.27413	31.20
51.1	1.23564	1.23782	27.86	57.6	1.27246	1.27471	31.25
51.2	1.23619	1.23838	27.91	57.7	1.27304	1.27529	31.30
51.3	1.23675	1.23894	27.96	57.8	1.27361	1.27587	31.35
51.4	1.23730	1.23949	28.02	57.9	1.27419	1.27645	31.40
51.5	1.23786	1.24005	28.07	58.0	1.27477	1.27703	31.46
51.6	1.23841	1.24060	28.12	58.1	1.27535	1.27761	31.51
51.7	1.23897	1.24116	28.17	58.2	1.27594	1.27819	31.56
51.8	1.23953	1.24172	28.23	58.3	1.27652	1.27878	31.61
51.9	1.24008	1.24228	28.28	58.4	1.27710	1.27936	31.66
52.0	1.24064	1.24284	28.33	58.5	1.27768	1.27994	31.71
52.1	1.24120	1.24339	28.38	58.6	1.27826	1.28052	31.76
52.2	1.24176	1.24395	28.44	58.7	1.27884	1.28111	31.82
52.3	1.24232	1.24451	28.49	58.8	1.27943	1.28169	31.87
52.4	1.24287	1.24507	28.54	58.9	1.28001	1.28228	31.92
52.5	1.24343	1.24563	28.59	59.0	1.28060	1.28286	31.97
52.6	1.24399	1.24619	28.65	59.1	1.28118	1.28345	32.02
52.7	1.24455	1.24675	28.70	59.2	1.28176	1.28404	32.07
52.8	1.24511	1.24731	28.75	59.3	1.28235	1.28462	32.13
52.9	1.24567	1.24788	28.80	59.4	1.28294	1.28520	32.18
53.0	1.24623	1.24844	28.86	59.5	1.28352	1.28579	32.23
53.1	1.24680	1.24900	28.91	59.6	1.28411	1.28638	32.28
53.2	1.24736	1.24956	28.96	59.7	1.28469	1.28697	32.33
53.3	1.24792	1.25013	29.01	59.8	1.28528	1.28755	32.38
53.4	1.24848	1.25069	29.06	59.9	1.28587	1.28814	32.43
53.5	1.24905	1.25126	29.12	60.0	1.28646	1.28873	32.49
53.6	1.24961	1.25182	29.17	60.1	1.28704	1.28932	32.54
53.7	1.25017	1.25238	29.22	60.2	1.28763	1.28991	32.59
53.8	1.25074	1.25295	29.27	60.3	1.28822	1.29050	32.64
53.9	1.25130	1.25351	29.32	60.4	1.28881	1.29109	32.69
54.0	1.25187	1.25408	29.38	60.5	1.28940	1.29168	32.74
54.1	1.25243	1.25465	29.43	60.6	1.28999	1.29227	32.79
54.2	1.25300	1.25521	29.48	60.7	1.29058	1.29286	32.85
54.3	1.25356	1.25578	29.53	60.8	1.29117	1.29346	32.90
54.4	1.25413	1.25635	29.59	60.9	1.29176	1.29405	32.95

TABLE 2—Continued

Degrees Brix or per cent sucrose by weight	Specific gravity at 20°/4° C	Specific gravity at 20°/20° C	Degrees Baumé (modulus 145)	Degrees Brix or per cent sucrose by weight	Specific gravity at 20°/4° C	Specific gravity at 20°/20° C	Degrees Baumé (modulus 145)
61.0	1.29235	1.29464	33.00	67.5	1.33163	1.33399	36.30
61.1	1.29295	1.29523	33.05	67.6	1.33225	1.33460	36.35
61.2	1.29354	1.29583	33.10	67.7	1.33287	1.33523	36.40
61.3	1.29413	1.29642	33.15	67.8	1.33348	1.33584	36.45
61.4	1.29472	1.29701	33.20	67.9	1.33410	1.33646	36.50
61.5	1.29532	1.29761	33.26	68.0	1.33472	1.33708	36.55
61.6	1.29591	1.29820	33.31	68.1	1.33534	1.33770	36.61
61.7	1.29651	1.29880	33.36	68.2	1.33596	1.33832	36.66
61.8	1.29710	1.29940	33.41	68.3	1.33658	1.33894	36.71
61.9	1.29770	1.29999	33.46	68.4	1.33720	1.33957	36.76
62.0	1.29829	1.30059	33.51	68.5	1.33782	1.34019	36.81
62.1	1.29889	1.30118	33.56	68.6	1.33844	1.34081	36.86
62.2	1.29948	1.30178	33.61	68.7	1.33906	1.34143	36.91
62.3	1.30008	1.30238	33.67	68.8	1.33968	1.34205	36.96
62.4	1.30068	1.30298	33.72	68.9	1.34031	1.34268	37.01
62.5	1.30127	1.30358	33.77	69.0	1.34093	1.34330	37.06
62.6	1.30187	1.30418	33.82	69.1	1.34155	1.34392	37.11
62.7	1.30247	1.30477	33.87	69.2	1.34217	1.34455	37.16
62.8	1.30307	1.30537	33.92	69.3	1.34280	1.34517	37.21
62.9	1.30367	1.30597	33.97	69.4	1.34342	1.34580	37.26
63.0	1.30427	1.30657	34.02	69.5	1.34405	1.34642	37.31
63.1	1.30487	1.30718	34.07	69.6	1.34467	1.34705	37.36
63.2	1.30547	1.30778	34.12	69.7	1.34530	1.34768	37.41
63.3	1.30607	1.30838	34.18	69.8	1.34592	1.34830	37.46
63.4	1.30667	1.30898	34.23	69.9	1.34655	1.34893	37.51
63.5	1.30727	1.30958	34.28	70.0	1.34717	1.34956	37.56
63.6	1.30787	1.31019	34.33	70.1	1.34780	1.35019	37.61
63.7	1.30848	1.31079	34.38	70.2	1.34843	1.35081	37.66
63.8	1.30908	1.31139	34.43	70.3	1.34906	1.35144	37.71
63.9	1.30968	1.31200	34.48	70.4	1.34968	1.35207	37.76
64.0	1.31028	1.31260	34.53	70.5	1.35031	1.35270	37.81
64.1	1.31088	1.31320	34.58	70.6	1.35094	1.35333	37.86
64.2	1.31149	1.31381	34.63	70.7	1.35157	1.35396	37.91
64.3	1.31209	1.31441	34.68	70.8	1.35220	1.35459	37.96
64.4	1.31270	1.31502	34.74	70.9	1.35283	1.35522	38.01
64.5	1.31330	1.31563	34.79	71.0	1.35346	1.35585	38.06
64.6	1.31391	1.31623	34.84	71.1	1.35409	1.35648	38.11
64.7	1.31452	1.31684	34.89	71.2	1.35472	1.35711	38.16
64.8	1.31512	1.31745	34.94	71.3	1.35535	1.35775	38.21
64.9	1.31573	1.31806	34.99	71.4	1.35598	1.35838	38.26
65.0	1.31633	1.31866	35.04	71.5	1.35661	1.35901	38.30
65.1	1.31694	1.31927	35.09	71.6	1.35724	1.35964	38.35
65.2	1.31755	1.31988	35.14	71.7	1.35788	1.36028	38.40
65.3	1.31816	1.32049	35.19	71.8	1.35851	1.36091	38.45
65.4	1.31877	1.32110	35.24	71.9	1.35914	1.36155	38.50
65.5	1.31937	1.32171	35.29	72.0	1.35978	1.36218	38.55
65.6	1.31998	1.32232	35.34	72.1	1.36041	1.36282	38.60
65.7	1.32059	1.32293	35.39	72.2	1.36105	1.36346	38.65
65.8	1.32120	1.32354	35.45	72.3	1.36168	1.36409	38.70
65.9	1.32181	1.32415	35.50	72.4	1.36232	1.36473	38.75
66.0	1.32242	1.32476	35.55	72.5	1.36295	1.36536	38.80
66.1	1.32304	1.32538	35.60	72.6	1.36359	1.36600	38.85
66.2	1.32365	1.32599	35.65	72.7	1.36423	1.36664	38.90
66.3	1.32426	1.32660	35.70	72.8	1.36486	1.36728	38.95
66.4	1.32487	1.32722	35.75	72.9	1.36550	1.36792	39.00
66.5	1.32548	1.32783	35.80	73.0	1.36614	1.36856	39.05
66.6	1.32610	1.32844	35.85	73.1	1.36678	1.36919	39.10
66.7	1.32671	1.32906	35.90	73.2	1.36742	1.36983	39.15
66.8	1.32732	1.32967	35.95	73.3	1.36805	1.37047	39.20
66.9	1.32794	1.33029	36.00	73.4	1.36869	1.37111	39.25
67.0	1.32855	1.33090	36.05	73.5	1.36933	1.37176	39.30
67.1	1.32917	1.33152	36.10	73.6	1.36997	1.37240	39.35
67.2	1.32978	1.33214	36.15	73.7	1.37061	1.37304	39.39
67.3	1.33040	1.33275	36.20	73.8	1.37125	1.37368	39.44
67.4	1.33102	1.33337	36.25	73.9	1.37189	1.37432	39.49

TABLE 2—Continued

Degrees Brix or per cent sucrose by weight	Specific gravity at 20° 4° C	Specific gravity at 20° 20° C	Degrees Baumé (modulus 145)	Degrees Brix or per cent sucrose by weight	Specific gravity at 20° 4° C	Specific gravity at 20°/20° C	Degrees Baumé (modulus 145)
74.0	1.37254	1.37496	39.54	80.5	1.41504	1.41754	42.71
74.1	1.37318	1.37561	39.59	80.6	1.41571	1.41821	42.76
74.2	1.37382	1.37625	39.64	80.7	1.41637	1.41888	42.81
74.3	1.37446	1.37689	39.69	80.8	1.41704	1.41955	42.85
74.4	1.37510	1.37754	39.74	80.9	1.41771	1.42022	42.90
74.5	1.37575	1.37818	39.79	81.0	1.41837	1.42088	42.95
74.6	1.37639	1.37883	39.84	81.1	1.41904	1.42155	43.00
74.7	1.37704	1.37947	39.89	81.2	1.41971	1.42222	43.05
74.8	1.37768	1.38012	39.94	81.3	1.42038	1.42289	43.10
74.9	1.37833	1.38076	39.99	81.4	1.42105	1.42356	43.14
75.0	1.37897	1.38141	40.03	81.5	1.42172	1.42423	43.19
75.1	1.37962	1.38206	40.08	81.6	1.42239	1.42490	43.24
75.2	1.38026	1.38270	40.13	81.7	1.42306	1.42558	43.29
75.3	1.38091	1.38335	40.18	81.8	1.42373	1.42625	43.33
75.4	1.38156	1.38400	40.23	81.9	1.42440	1.42692	43.38
75.5	1.38220	1.38465	40.28	82.0	1.42507	1.42759	43.43
75.6	1.38285	1.38530	40.33	82.1	1.42574	1.42827	43.48
75.7	1.38350	1.38595	40.38	82.2	1.42642	1.42894	43.53
75.8	1.38415	1.38660	40.43	82.3	1.42709	1.42961	43.57
75.9	1.38480	1.38725	40.48	82.4	1.42776	1.43029	43.62
76.0	1.38545	1.38790	40.53	82.5	1.42844	1.43096	43.67
76.1	1.38610	1.38855	40.57	82.6	1.42911	1.43164	43.72
76.2	1.38675	1.38920	40.62	82.7	1.42978	1.43231	43.77
76.3	1.38740	1.38985	40.67	82.8	1.43046	1.43298	43.81
76.4	1.38805	1.39050	40.72	82.9	1.43113	1.43366	43.86
76.5	1.38870	1.39115	40.77	83.0	1.43181	1.43434	43.91
76.6	1.38935	1.39180	40.82	83.1	1.43248	1.43502	43.96
76.7	1.39000	1.39246	40.87	83.2	1.43316	1.43569	44.00
76.8	1.39065	1.39311	40.92	83.3	1.43384	1.43637	44.05
76.9	1.39130	1.39376	40.97	83.4	1.43451	1.43705	44.10
77.0	1.39196	1.39442	41.01	83.5	1.43519	1.43773	44.15
77.1	1.39261	1.39507	41.06	83.6	1.43587	1.43841	44.19
77.2	1.39326	1.39573	41.11	83.7	1.43654	1.43908	44.24
77.3	1.39392	1.39638	41.16	83.8	1.43722	1.43976	44.29
77.4	1.39457	1.39704	41.21	83.9	1.43790	1.44044	44.34
77.5	1.39523	1.39769	41.26	84.0	1.43858	1.44112	44.38
77.6	1.39588	1.39835	41.31	84.1	1.43926	1.44180	44.43
77.7	1.39654	1.39901	41.36	84.2	1.43994	1.44249	44.48
77.8	1.39719	1.39966	41.40	84.3	1.44062	1.44317	44.53
77.9	1.39785	1.40032	41.45	84.4	1.44130	1.44385	44.57
78.0	1.39850	1.40098	41.50	84.5	1.44198	1.44453	44.62
78.1	1.39916	1.40164	41.55	84.6	1.44266	1.44521	44.67
78.2	1.39982	1.40230	41.60	84.7	1.44334	1.44590	44.72
78.3	1.40048	1.40295	41.65	84.8	1.44402	1.44658	44.76
78.4	1.40113	1.40361	41.70	84.9	1.44470	1.44726	44.81
78.5	1.40179	1.40427	41.74	85.0	1.44539	1.44794	44.86
78.6	1.40245	1.40493	41.79	85.1	1.44607	1.44863	44.91
78.7	1.40311	1.40559	41.84	85.2	1.44675	1.44931	44.95
78.8	1.40377	1.40625	41.89	85.3	1.44744	1.45000	45.00
78.9	1.40443	1.40691	41.94	85.4	1.44812	1.45068	45.05
79.0	1.40509	1.40758	41.99	85.5	1.44881	1.45137	45.09
79.1	1.40575	1.40824	42.03	85.6	1.44949	1.45205	45.14
79.2	1.40641	1.40890	42.08	85.7	1.45018	1.45274	45.19
79.3	1.40707	1.40956	42.13	85.8	1.45086	1.45343	45.24
79.4	1.40774	1.41023	42.18	85.9	1.45154	1.45411	45.28
79.5	1.40840	1.41089	42.23	86.0	1.45223	1.45480	45.33
79.6	1.40906	1.41155	42.28	86.1	1.45292	1.45549	45.38
79.7	1.40972	1.41222	42.32	86.2	1.45360	1.45618	45.42
79.8	1.41039	1.41288	42.37	86.3	1.45429	1.45686	45.47
79.9	1.41105	1.41355	42.42	86.4	1.45498	1.45755	45.52
80.0	1.41172	1.41421	42.47	86.5	1.45567	1.45824	45.57
80.1	1.41238	1.41488	42.52	86.6	1.45636	1.45893	45.61
80.2	1.41304	1.41554	42.57	86.7	1.45704	1.45962	45.66
80.3	1.41371	1.41621	42.61	86.8	1.45773	1.46031	45.71
80.4	1.41437	1.41688	42.66	86.9	1.45842	1.46100	45.75

TABLE 2—Continued

Degrees Brix or per cent sucrose by weight	Specific gravity at 20°/4° C	Specific gravity at 20°/20° C	Degrees Baumé (modulus 145)	Degrees Brix or per cent sucrose by weight	Specific gravity at 20°/4° C	Specific gravity at 20°/20° C	Degrees Baumé (modulus 145)
87.0	1.45911	1.46170	45.80	93.5	1.50472	1.50738	48.81
87.1	1.45980	1.46239	45.85	93.6	1.50543	1.50810	48.85
87.2	1.46050	1.46308	45.89	93.7	1.50615	1.50881	48.90
87.3	1.46119	1.46377	45.94	93.8	1.50686	1.50952	48.94
87.4	1.46188	1.46446	45.99	93.9	1.50757	1.51024	48.99
87.5	1.46257	1.46516	46.03	94.0	1.50829	1.51096	49.03
87.6	1.46326	1.46585	46.08	94.1	1.50900	1.51167	49.08
87.7	1.46395	1.46654	46.13	94.2	1.50972	1.51239	49.12
87.8	1.46464	1.46724	46.17	94.3	1.51044	1.51311	49.17
87.9	1.46534	1.46793	46.22	94.4	1.51115	1.51382	49.22
88.0	1.46603	1.46862	46.27	94.5	1.51187	1.51454	49.26
88.1	1.46673	1.46932	46.31	94.6	1.51258	1.51526	49.31
88.2	1.46742	1.47002	46.36	94.7	1.51330	1.51598	49.35
88.3	1.46812	1.47071	46.41	94.8	1.51402	1.51670	49.40
88.4	1.46881	1.47141	46.45	94.9	1.51474	1.51742	49.44
88.5	1.46950	1.47210	46.50	95.0	1.51546	1.51814	49.49
88.6	1.47020	1.47280	46.55	95.1	1.51617	1.51886	49.53
88.7	1.47090	1.47350	46.59	95.2	1.51689	1.51958	49.58
88.8	1.47159	1.47420	46.64	95.3	1.51761	1.52030	49.62
88.9	1.47229	1.47489	46.69	95.4	1.51833	1.52102	49.67
89.0	1.47299	1.47559	46.73	95.5	1.51905	1.52174	49.71
89.1	1.47368	1.47629	46.78	95.6	1.51977	1.52246	49.76
89.2	1.47438	1.47699	46.83	95.7	1.52049	1.52318	49.80
89.3	1.47508	1.47769	46.87	95.8	1.52121	1.52390	49.85
89.4	1.47578	1.47839	46.92	95.9	1.52193	1.52463	49.90
89.5	1.47648	1.47909	46.97	96.0	1.52266	1.52535	49.94
89.6	1.47718	1.47979	47.01	96.1	1.52338	1.52607	49.98
89.7	1.47788	1.48049	47.06	96.2	1.52410	1.52680	50.03
89.8	1.47858	1.48119	47.11	96.3	1.52482	1.52752	50.08
89.9	1.47928	1.48189	47.15	96.4	1.52555	1.52824	50.12
90.0	1.47998	1.48259	47.20	96.5	1.52627	1.52897	50.16
90.1	1.48068	1.48330	47.24	96.6	1.52699	1.52969	50.21
90.2	1.48138	1.48400	47.29	96.7	1.52772	1.53042	50.25
90.3	1.48208	1.48470	47.34	96.8	1.52844	1.53114	50.30
90.4	1.48278	1.48540	47.38	96.9	1.52917	1.53187	50.34
90.5	1.48348	1.48611	47.43	97.0	1.52989	1.53260	50.39
90.6	1.48419	1.48681	47.48	97.1	1.53062	1.53332	50.43
90.7	1.48489	1.48752	47.52	97.2	1.53134	1.53405	50.48
90.8	1.48559	1.48822	47.57	97.3	1.53207	1.53478	50.52
90.9	1.48630	1.48893	47.61	97.4	1.53279	1.53551	50.57
91.0	1.48700	1.48963	47.66	97.5	1.53352	1.53623	50.61
91.1	1.48771	1.49034	47.71	97.6	1.53425	1.53696	50.66
91.2	1.48841	1.49104	47.75	97.7	1.53498	1.53769	50.70
91.3	1.48912	1.49175	47.80	97.8	1.53570	1.53842	50.75
91.4	1.48982	1.49246	47.84	97.9	1.53643	1.53915	50.79
91.5	1.49053	1.49316	47.89	98.0	1.53716	1.53988	50.84
91.6	1.49123	1.49387	47.94	98.1	1.53789	1.54061	50.88
91.7	1.49194	1.49458	47.98	98.2	1.53862	1.54134	50.93
91.8	1.49265	1.49529	48.03	98.3	1.53935	1.54207	50.97
91.9	1.49336	1.49600	48.08	98.4	1.54008	1.54280	51.02
92.0	1.49406	1.49671	48.12	98.5	1.54081	1.54353	51.06
92.1	1.49477	1.49741	48.17	98.6	1.54154	1.54426	51.10
92.2	1.49548	1.49812	48.21	98.7	1.54227	1.54499	51.15
92.3	1.49619	1.49883	48.26	98.8	1.54300	1.54573	51.19
92.4	1.49690	1.49954	48.30	98.9	1.54373	1.54646	51.24
92.5	1.49761	1.50026	48.35	99.0	1.54446	1.54719	51.28
92.6	1.49832	1.50097	48.40	99.1	1.54519	1.54793	51.33
92.7	1.49903	1.50168	48.44	99.2	1.54593	1.54866	51.37
92.8	1.49974	1.50239	48.49	99.3	1.54666	1.54939	51.42
92.9	1.50045	1.50310	48.53	99.4	1.54739	1.55013	51.46
93.0	1.50116	1.50381	48.58	99.5	1.54813	1.55087	51.50
93.1	1.50187	1.50453	48.62	99.6	1.54886	1.55160	51.55
93.2	1.50258	1.50524	48.67	99.7	1.54960	1.55234	51.59
93.3	1.50329	1.50595	48.72	99.8	1.55033	1.55307	51.64
93.4	1.50401	1.50667	48.76	99.9	1.55106	1.55381	51.68
				100.0	1.55180	1.55454	51.73

WASHINGTON : GOVERNMENT PRINTING OFFICE : 1918

DEPARTMENT OF COMMERCE

TECHNOLOGIC PAPERS

OF THE

BUREAU OF STANDARDS

S. W. STRATTON, DIRECTOR

No. 116

SILICA REFRACTORIES

FACTORS AFFECTING THEIR QUALITY AND METHODS OF TESTING THE RAW MATERIALS AND FINISHED WARE

BY

DONALD W. ROSS, Assistant Ceramic Chemist

Bureau of Standards

ISSUED APRIL 19, 1919

PRICE, 20 CENTS

Sold only by the Superintendent of Documents, Government Printing Office
Washington, D. C.

WASHINGTON
GOVERNMENT PRINTING OFFICE
1919

SILICA REFRACTORIES—FACTORS AFFECTING THEIR QUALITY AND METHODS OF TESTING THE RAW MATERIALS AND FINISHED WARE

By Donald W. Ross

CONTENTS

	Page
I. Introduction	4
II. Review of previous work	4
III. General résumé of data relating to materials, manufacture, and uses	10
1. Nature of silica refractories	10
2. Geology	14
3. Manufacture	20
4. Uses in industries	23
IV. Statement of work done and description of methods	25
1. General statement	25
2. Heating tests	25
(*a*) Series 1	25
(*b*) Series 2	26
(*c*) Series 3	27
(*d*) Series 4	27
(*e*) Series 5	28
(*f*) Series 6	29
(*g*) Series 7	30
(*h*) Series 8	30
3. Properties of finished bricks and data on manufacture	30
(*a*) Data on commercial bricks	30
(*b*) Analyses and softening temperatures	31
(*c*) Load tests and volume changes at 300° C	31
(*d*) Micro studies	31
(*e*) Effect of slags	31
(*f*) Screen analyses	31
(*g*) Cold strength tests	31
V. Results of work and discussion of same	32
1. Heating tests	32
(*a*) Series 1	32
(*b*) Series 2	33
(*c*) Series 3	36
(*d*) Series 4	38
(*e*) Series 5	43
(*f*) Series 6	45
(*g*) Series 7	47
(*h*) Series 8	48
2. Properties of finished bricks and data on manufacture	48
(*a*) Data on commercial bricks	48
(*b*) Analyses and softening temperatures	53
(*c*) Load tests	55
(*d*) Micro studies	57
(*e*) Effect of slags	60
(*f*) Screen analyses	61
(*g*) Cold strength tests	64

Page

VI. General results 65
1. Composition 66
2. Micro studies 66
(*a*) Raw material 66
(*b*) Burned material 67
3. Density 68
4. Volume and porosity changes 69
(*a*) Permanent changes 69
(*b*) Thermal changes 70
5. Mechanical strength 72
(*a*) Mechanical strength, raw 72
(*b*) Mechanical strength, burned 74
6. Slag resistance 76
7. Special problems 76
8. Testing and inspecting materials and ware 77
(*a*) Raw materials 77
(*b*) Finished ware 78
VII. Summary 81

I. INTRODUCTION

The tremendous trade in silica refractories for by-product coke ovens, steel and copper reverberatory furnaces, etc., which has sprung up in the last two years has caused attention to be turned to the need of complete information concerning the manufacture and properties of these materials and of specifications for their inspection.

Although considerable valuable research work has already been done in connection with silica refractories, it was thought desirable to review the information at hand and to do experimental work upon points which had not previously been as thoroughly worked out as would seem necessary.

II. REVIEW OF PREVIOUS WORK

In the study of previous work on silica refractories we find that the work of the Geophysical Laboratory of the Carnegie Institution [1] in connection with the physico-crystalline relations of the forms of silica and the silicates of the ternary system CaO–Al_2O_3–SiO_2 serves at once as a review and summary of most work on these subjects to date. Likewise, the work of McDowell [2] on the silica refractories may be considered as a practical summation of the previously published data on manufactured silica refractories. In this work is also presented a bibliography of the literature covering both the silica minerals and the manufactured ware.

[1] Stability Relations of the Silica Minerals, C. N. Fenner, Am. J. S., 36; October, 1913. The Ternary system CaO–Al_2O_3–SiO_2, Am. J. S., 39; January, 1915.

[2] A Study of the Silica Refractories, J. Spotts McDowell, Am. Inst. Mining Engineers, Bull. 119.

Hence, the individual references are not taken up here, except some that refer to varieties of raw silica brick materials and other data which have not received particular emphasis by previous writers. Such points of the above and later work are here considered as appear to have special bearing upon the present work.

In some cases the silica rock as mined is either hewn into blocks or crushed and used directly in furnace construction. Richter [3] states that Crummendorf schists which are very fine grained and exceptionally uniform in structure and impurities and contain from 95 to 96 per cent of silica are furnished in rough or sawn blocks. These are used directly as refractories in place of clay fire brick and are supposed to last longer. Some varieties contract in use; others expand slightly.

In the choice of raw materials for the manufacture of silica refractories, Khoetsky [4] has considered the subject from the standpoint of chemical purity, size of grain obtainable, and the magnitude of the increase in volume on heating. He concludes that to meet the temperature requirements in use the brick must not contain less than 96 per cent silica (SiO_2) (98 per cent silica for the raw material); that the rock must not yield too fine a powder on crushing; and, third, the order of merit of the natural forms of silica is given as chalcedony, old quartzites, and vein quartz. Quartz schists, sandstones, and sand are considered unsuitable, the first on account of their structure and the presence of many impurities in the form of inclusions, the two latter on account of their variability in composition and their excessive fineness after grinding.

The same problem has been attacked by determining the specific gravity [5] of the material before and after repeated heatings to cone 15–16 (1450° C approximately). The author designates (*a*) as good material, (*b*) as fair, and (*c*) as poor, and presents the following figures in Table 1:

TABLE 1.—Differences in Rate of Drop in Specific Gravity of Different Varieties of Silica Raw Materials on Being Heated Repeatedly to Cones 15–16

Heatings to cone 15–16	Specific gravity		
	(a)	(b)	(c)
Before heating	2.65	2.65	2.64
After first heating	2.40	2.47	2.53
After third heating	2.38	2.37	2.51
After fifth heating	2.38	2.37	2.43

NOTE.—(*a*) Transformed most readily, (*b*) required a somewhat greater heat treatment, and (*c*) had apparently not reached minimum attainable specific gravity by the end of the test.

[3] Hugo Richter, Zs. Ver. Zucherind, **65**, pp. 263–273; 1915.
[4] Rev. Soc. Russe de Metall, **1**, pp. 170–202; 1914.
[5] Anon, Tonind. Ztg., **38**, pp. 163–165.

As seen from the figures, (*a*) practically completes its drop in specific gravity in one burn while (*c*) continues to drop for a long series of ignitions.

Concerning the rates of transformation of the different forms of silica to cristobalite on being heated, Endell and Rieke [6] state that when heated to 1400° C rock crystal was slowest in being transformed, while strongly twinned pseudomorphic quartz from the Taunus and pegmatitic quartz from Norway were found to be largely converted in two or three such heatings. Powdered silica glass, amorphous silica, and chalcedony were completely changed

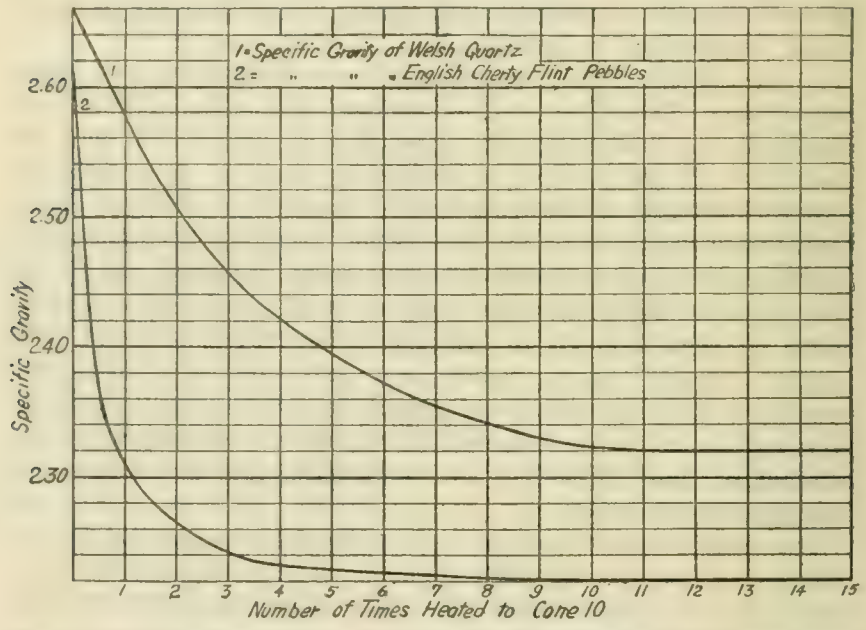

1 = Specific Gravity of Welsh Quartz
2 = " " " English Cherty Flint Pebbles
2.60
2.50
2.40
2.30
Specific Gravity
1
2
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15
Number of Times Heated to Cone 10

FIG. 1.—*Rate of drop in specific gravity of English flint pebbles and Welsh quartz, when repeatedly heated to cone 10 (after Mellor)*

after a single burning. The rate of transformation of silica glass into cristobalite was found to increase rapidly with the temperature from 1300° C upward.

In Fig. 1 are shown the results of work by Mellor [7] on the changes in specific gravity of quartz and English flint after repeated heatings to cone 10. The quartz was presumably Welsh and the flint was derived from flint pebbles of the chalky limestones of southeastern England or northern France. These flint pebbles are cryptocrystalline silica and their origin is practically identical with that

[6] K. Endell and R. Rieke, Zs. Angew. Chem., 25, pp. 2019–2020; 1912.
[7] J. W. Mellor and A. J. Campbell, Trans. Eng. Ceramic Soc., 15, pp. 77–116; 1915–16.

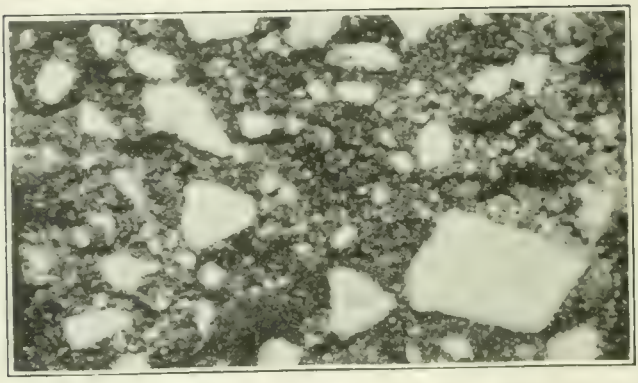


FIG. 2. *Photomicrograph of polished surface of brick made from regular grind, × 2; Medina quartzite (after McDowell)*

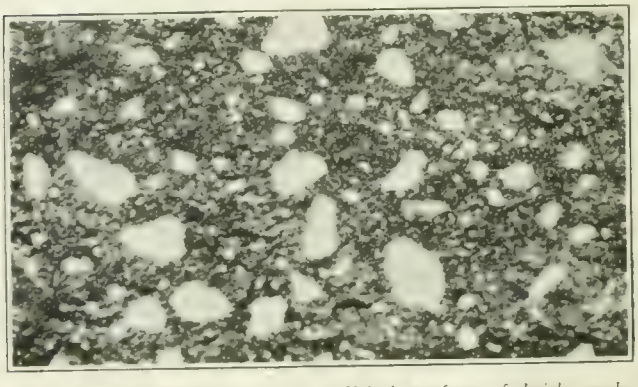


FIG. 3. *Photomicrograph of polished surface of brick made from shape grind, × 2; Medina quartzite (after McDowell)*

of chert. As shown in this article, the flint pebbles have a lower original specific gravity than quartz, are converted to a lower specific gravity more rapidly than quartz at this temperature, and the final specific gravity is less than that of the resultant product from quartz.

In connection with load tests on silica brick, the work of Desher and Fulweiler [8] shows that with a heating rate of approximately 450° C per hour, the specimen being tested expands decidedly during the first two hours and is then practically constant in size until a temperature of approximately 1400° C is reached, above which there is apt to be further expansion. A comparison of this high temperature expansion with the permanent expansion of the piece indicates that it (high temperature expansion) is due to permanant change rather than thermal expansion, and that in the case of underburned material it will be considerable, while with well-burned material it will be small.

The screen analyses of the materials used by McDowell [9] in his work are shown in Table 2.

TABLE 2.—Screen Analyses of Raw Commercial Silica Brick Mixes as Given by McDowell

Grind	Percentage held on screen					Per cent through 40-mesh
	8-mesh	16-mesh	20-mesh	30-mesh	40-mesh	
Regular	15.3	13.4	4.6	5.4	4.8	56.5
Medium or shape	10.2	13.1	2.2	5.0	4.7	64.8
Fine	----------	14.3	4.1	6.5	4.1	71.0

NOTE.—"Regular" is the grind used in making standard 9-inch bricks; "medium or shape" is the grind used in making difficult shapes; "fine" is not usually used commercially.

The specific volumes of the different forms of silica occurring in silica refractories, as given by him, are taken up below under "Nature of silica refractories." The materials used by him were made from Medina quartzite. The coarsest grind corresponds to that usually used in standard 9-inch brick. The medium grind is that which is usually employed in making shapes. The fine grind is material which, after passing through an 8-mesh sieve, was returned to the grinding pan and reground long enough to add the lime and mix it in thoroughly. Figs. 2 and 3 show sections of brick made from these grinds. These sections well illustrate the proportion of coarse to fine materials.

[8] Report of The Committee on Refractories, Am. Gas. Inst.; 1915. [9] Loc. cit.

In crushing tests he has expressed the mean compressive strength minus the average deviation as the effective ultimate strength, and likewise for transverse tests the effective ultimate strength is given as the mean modulus of rupture minus the average deviation in moduli. His data show that in compression the fine grind was the strongest and the regular grind the weakest. In the cross breaking tests the fine grind shows little if any advantage over the regular grind, while the medium grind is considerably stronger. On reburning, both compressive and transverse strengths were found to increase for all grinds until a maximum was reached in each case, after which further burnings tended to cause both compressive and transverse strength to decrease. McDowell's opinion[10] is that this decrease in strength was caused by cracks, which were occasioned by the repeated heating and cooling.

He carried out spalling tests as follows: The bricks to be tested were heated at the rate of 15° C per hour to 600° C and held at that temperature for three hours, after which they were withdrawn and placed on steel pallets away from drafts and allowed to cool. When cold they were tested and their crushing and transverse strengths were compared with those of bricks from the same lot, but which had not received this heat treatment. The results show that on repeated burning as the percentage of cristobalite in the bricks decreased and the percentage of tridymite increased the spalling tendency decreased.

Nesbitt and Bell [11] conducted a series of tests on silica brick which were specially made from Pennsylvania ganister. Three lots of rock were used which were ground dry to pass through 12, 8, and 4 mesh screens, respectively. Two per cent of lime and 9 per cent of water were mixed with the ground quartzite. These mixes were then made up into standard 9-inch brick under various pressures for each mesh. When dry the brick were given a regular silica-brick burn.

Impact, spalling, and slagging tests were made upon these brick. The impact test was conducted by placing a heated brick on end on a steel block and dropping a steel ball upon the center of the other end. The spalling test was conducted by heating a weighed brick on one end to 540° C and then plunging the heated end into water. Ten such treatments constituted a test, after which

[10] Privately communicated.

[11] C. E. Nesbitt and M. L. Bell, Proc. Am. Soc. for Testing Materials; 1917.

the bricks were dried at 100° C; all loose material removed; the remainder weighed, and the loss given in per cent of the original weight. The slagging test was conducted by heating a brick, containing a cavity of known cross section, to 1350° C in a furnace and placing in the cavity a definite amount of slag of known composition. The temperature was held at 1350° C for two hours from the time the slag was added, after which the furnace was permitted to cool. When cold the brick was sawn in two through the center of the cavity. By means of a planimeter the area of the slag penetration was measured, and from this was subtracted the cross sectional area of the original cavity. Nesbitt and Bell's figures indicate that increased pressure in molding, between 187 and 2500 pounds per square inch, is accompanied by a continuous increase in resistance to impact; that slag penetration increases with increased size of grain; and that the spalling tendency decreases with increased size of grain. In summing up their work they conclude that as a whole the coarsest grained material used by them gave the best results.

TABLE 3.—Crushing Strengths of "Star" Silica Brick at Various Temperatures

Temperature in degrees centigrade	Crushing strength of hot brick		Temperature in degrees centigrade	Crushing strength of hot brick	
	Kilograms per square centimeter	Pounds per square inch		Kilograms per square centimeter	Pounds per square inch
15	170	2418	1200	85	1209
520	158	2247	1320	62	882
670	150	2133	1460	50	711
800	139	1977	1540	37	526
950	125	1778	1600	30	427
1050	120	1707	1700	12	[a]171

[a] Extrapolated.

In Table 3 are presented data of crushing tests on Star Silica brick at various temperatures, as reported by Le Chatelier.[12] The 1700° C figure has been extrapolated by a continuation of the curve.

[12] Revue de Metallurgie; June, 1917.

III. GENERAL RÉSUMÉ OF DATA RELATING TO MATERIALS, MANUFACTURE, AND USES

1. NATURE OF SILICA REFRACTORIES

It is evident that silica refractories must act primarily as does SiO_2. The stability relations of the different crystal modifications of SiO_2 as determined by the Geophysical Laboratory of the Carnegie Institution, briefly summarized are:

Inversion	Temperature	Remarks
α quartz—β quartz	575° C	Rapid, reversible.
β quartz—β_2 tridymite	870° C±10° C	Very sluggish, reversible.
β_2 tridymite—β cristobalite	1470° C±10° C	Do.
α tridymite—β_1 tridymite	117° C	Rapid, reversible.
β_1 tridymite—β_2 tridymite	163° C	Do.
α cristobalite—β cristobalite	220–275° C[a]	Do.

[a] Temperature depends on previous heat treatment; probably monotropic. Changes slowly to chalcedony, tridymite, or quartz in the presence of a flux at relatively low tempertures.

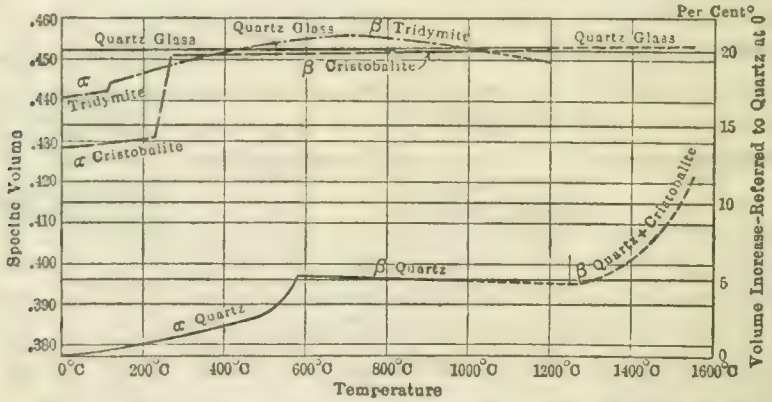


FIG. 4.—*Specific volumes of the silica minerals and quartz glass (arranged by McDowell).*

Cristobalite was formerly believed to melt to a silica glass at approximately 1625° C. More recent investigations have shown that its melting temperature under certain conditions is considerably above this figure.

Silica crystallizing from molten silicate magmas forms practically pure crystals, except that they may contain very small quantities of calcium silicate in solid solution.

"If either amorphous modification (silica glass or precipitated silica) is heated without a flux at 1300 ° C or 1400° C cristobalite

alone is obtained, although the temperature is within the range of tridymite. The process halts at the cristobalite stage, and can only be carried to completion by the addition of a flux. Similarly, ground quartz heated without a flux at high temperatures but still below the 1470° C inversion point is changed to cristobalite and not tridymite which might be expected." [13] A large pure quartz crystal when similarly heated without a flux will be converted on the surface and along cracks to cristobalite, which on cooling forms a white coating over the clear unaltered quartz of the crystal, and tends to spall off because of the Alpha-Beta cristobalite inversion.

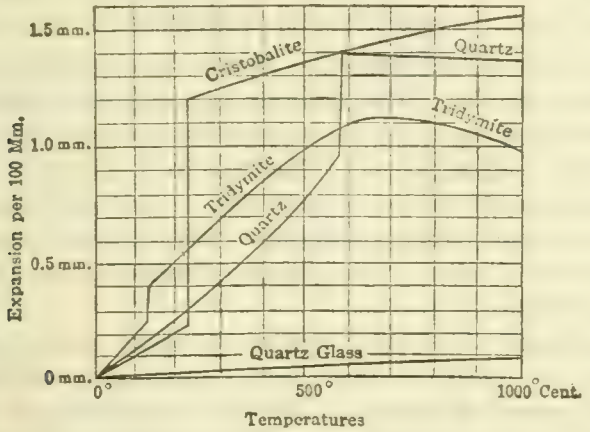


FIG. 5.—*Results of Le Chatelier's experiments in thermal expansion.*[a]

In Figs. 4 and 5 are shown the specific volumes of the different silica minerals and of silica glass and the thermal expansion of these minerals according to Le Chetelier's experiments. Results given by McDowell and others on the quantitative microscopic determinations of the various crystal forms of silica in silica refractories made from quartzites, indicate that the inversion of quartz to cristobalite takes place, but that with long continued heating tridymite eventually makes its appearance after a large percentage of the quartz has already been converted into cristobalite. Table 4 shows the micro-analyses of brick made from Medina quartzite which were repeatedly burned in a commercial kiln to cone 14–15.

[a] Le Chatelier; La Silica, Revue, 1, p. 90; 1913.

[13] C. N. Fenner, Stability Relations of The Silica Minerals, Am. Jour. Soc. 36, p. 339.

TABLE 4.—Percentages of Quartz Plus Silicates, Cristobalite, and Tridymite present in Silica Brick Made from Medina Quartzite after Repeated Heatings in a Commercial Kiln to Cones 14–15

Number of burn	Percentage by volume		
	Quartz plus silicates	Cristobalite	Tridymite
1	25	71	4
3	14	64	20
4	12	58	30
6	14	42	44
8	14	33	53
10	11	28	61

NOTE.—Results on burns Nos. 6, 8, and 10 privately communicated by McDowell.

The figures well illustrate the change of quartz through cristobalite to tridymite. The indications are that for this material and method of heating the final equilibrium would be as follows: The quartz + silicates would decrease to 13 per cent, at which point there would be no quartz left. The cristobalite would entirely disappear and the tridymite content would hence rise to 87 per cent as a limit. The material of burn No. 10 is the same as No. 6 of Table 16.

In the system CaO–SiO_2,[14] where a large excess of SiO_2 is present, as is the case with silica refractories, the lime-silica compound formed is probably $CaOSiO_2$. α $CaOSiO_2$ is the stable form from 1200° C to its melting temperature 1540° ± 2°C. Below 1200° C β $CaOSiO_2$ is the stable form. This inversion at 1200° C may possibly have a slight tendency to disrupt the ware, but the quantity of this material present, if any, is so small that it can hardly be expected to have any pronounced effect. The eutectic between $CaOSiO_2$ and silica has the weight percentage composition, CaO 37, SiO_2 63; its melting temperature is 1436° ± 2° C. In the neighborhood of pure SiO_2, however, and for some 8 to 10 per cent by weight composition from pure silica the melting temperature does not drop much below 1600° C.

In the system Al_2O_3–SiO_2 the low eutectic has the weight percentage composition Al_2O_3 13, SiO_2 87 and melts at 1610° C. In the neighborhood of pure silica, however, and up to the limits of Al_2O_3 (0.5 per cent) occurring in silica refractories, the melting temperature is close to that of pure SiO_2. In the ternary system CaO–Al_2O_3–SiO_2 the region near pure SiO_2 shows that for the

[14] Data taken from Am. J. S., 39; January, 1915 The Ternary System CaO–Al_2O_3–SiO_2.

percentages of CaO and Al_2O_3 under consideration, the melting point gradually increases as we pass from CaO to Al_2O_3.

Data of the Geophysical Laboratory published and unpublished indicate that iron oxide which may be scattered over the surface of the grains of a quartzite as hydrated oxide, gathers into grains of hematite (Fe_2O_3) when heated to comparatively low temperatures. The hematite so formed dissociates slightly, even at 1100° C and under the oxygen pressure of the air (152 mm of mercury), forming a solid solution of magnetite (Fe_3O_4) in hematite. As the temperature is raised more oxygen is evolved from this solid solution and finally the composition of Fe_3O_4 is reached at 1580° C, at which temperature melting of pure magnetite takes place. These grains of magnetite may combine with adjacent silica grains, if subjected to a reducing atmosphere, to form local spots of iron-bearing silica glass. Other ingredients, such as CaO for instance, in conjunction with the iron oxide, may tend to cause softening of the quartzite at a comparatively low temperature. It has been shown that the eutectics (points of maximum fusibility) in the system CaO–Fe_2O_3 form exceedingly fluid liquids and are at approximately 1200° C, and consequently the effect of a few per cent of iron oxide and lime on the softening of a brick may be very great. This effect may be minimized by other factors, such as lack of contact, etc. With pure silica in contact with iron oxide the effect is more difficult to predict, as this system has not been worked out. The data recorded below indicate, however, that the effect is much less than is ordinarily supposed.

The work of Fieldner [15] and others in connection with the effect of iron oxide upon the fusion temperatures of kaolin + CaO, and coal ash + CaO indicates that up to approximately 5 per cent Fe_2O_3 the iron had but little effect in reducing the softening temperatures of the mixtures, and that for this amount of iron there was very little difference in the fusion temperatures whether oxidizing or reducing conditions prevailed.

The most refractory silica brick when made into cones and heated rapidly, as described later, soften and bend over at a slightly higher temperature than that at which Orton cone 32 bends over. Kanolt[16] says: "pure silica melts at about 1600° C.[17] However,

[15] Technologic Paper No. 154, U. S. Bureau of Mines, as yet unpublished. Effect of Iron Compounds on the Softening Temperature of Coal Ash, Kaolin, and Kaolin-Lime Mixtures, in various atmospheres.

[16] Technologic Paper No. 10, this Bureau, p. 14, Melting Points of Fire Brick.

[17] This temperature was later corrected to approximately 1625° C as given above, and more recently to 1715±10° C.

the fused silica possesses such extreme viscosity near the melting point that it does not flow or change shape distinctly until considerably higher temperatures are reached. I have obtained 1750° C as the apparent melting point of pure silica; i. e., the temperature at which it flows distinctly. This temperature, however, is naturally a very indefinite one. The temperature at which silica brick flow distinctly is more definite.

2. GEOLOGY

The matter presented under this heading is intended to indicate what some of the chief varieties of material are, which have been used for silica refractories, and in general how they occur. For as new deposits are opened up it is probable that their value can be more or less predicted by such knowledge.

It is probable that most silica brick in all countries have been manufactured from the older quartzites. Although amorphous silica, chert, chalcedony, vein quartz, and quartz schist have been used to some extent, Khoetsky [18] suggests that the order of merit for silica refractories is chalcedony, old quartzites, and vein quartz, and also that quartz schist, sandstone, and sand are unsuitable for the manufacture of brick, the first on account of its structure and the presence of many impurities in the form of inclusions; the two latter on account of their variability of composition and their excessive fineness after being ground.

In the United States quartzite is the usual material, although on a small scale silica sand cemented with chalcedony and minute quartz crystals, and a pure rock chert are used, and in one case a small percentage of a material consisting of quartz pebbles cemented together by chalcedony is introduced into the brick mix.

Quartzites are sedimentary deposits and consist of silica sand, cemented together by silica. In some cases the grains still show a rounded water-worn form, while in more highly metamorphosed varieties the additional precipitated silica has built up the grains so that they are angular and tightly interlocking, thus forming a rock of low porosity. The metamorphosis in most cases is probably caused both by heat and by the deposition of silica from percolating waters containing it. Quartz schist results from quartzite when the alteration or metamorphosis is accompanied by mechanical deformation, by which a cleavage or schistose structure is developed.

[18] Khoetsky, A. K., Rev. Soc. Russe de Metall., I, pp. 170–202; 1914.

Chert is sedimentary and is laid down from siliceous sponges, etc. It is frequently found interbedded with limestone. It is cryptocrystalline, showing slight crystal formation in a mass of amorphous material. It is evidently amorphous when first laid down. Probably some of it is opaline, as there usually appear to be small amounts of water combined with the silica.

Chalcedony may be laid down from water solutions at comparatively low temperatures, even down to atmospheric temperature. Quartz crystals also frequently form, admixed with the chalcedony.

With reference to the sources of silica brick materials in Great Britain, the following statements are taken from a paper by Bywater:[19]

The principal sources of this (silica brick material) are in the neighborhood of Neath Valley in the south of Wales, and the "black" or quartz ganister found in the neighborhood of Sheffield; * * * they have very similar composition. Samples of raw material have been found to contain:

	Dinas quartz	Black Ganister		Dinas quartz	Black Ganister
SiO_2	97.6	98.5	CaO	0.2	0.2
Al_2O_3	.5	.3	K_2O	.09	Trace
Fe_2O_3	1.5	1.3	Na_2O	.03	Trace

The South Wales or Dinas quartz has a pale grayish blue tinge and is worked in open quarries about 50 to 60 feet high with thin veins of yellow clay between. The rock is hard, and steam or compressed air drills are used. Very little impurity is found, and the rock is crushed and mixed with 1 to 1.5 per cent of lime and a small percentage of siliceous clay to toughen the brick. * * * No calcined material is used. The brick are burned at about 2600° F (1427° C) and expand somewhat on burning and subsequent heating.

"*Black*" *Ganister*.—This forms the seat stone (floor) beneath certain seams in the lower coal measures near Sheffield. It is mined by means of rock drills; and the method of manufacture is similar to that of the Dinas quartz brick, except that there are some fairly well-defined impurities found in the raw material which can be picked out. There is also usually an admixture of 25 per cent of calcined material.

The Millstone grit formation, which lies below the coal measures of the carboniferous age occurs in quantity near Sheffield. In some cases blocks of this have been used in furnaces with fair results.

Over half the rock used in the United States for silica refractories is derived from the Tuscarora (more popularly known as Medina) formation, and comes almost entirely from Blair and Huntingdon Counties, Pa. This formation consists of two distinct divisions and is immediately underlain by the Juniata forma-

[19] Frederick J. Bywater, reprint in Trans. of The Faraday Society, 12; June, 1917.

tion. (The Oneida of the Second Geological Survey of Pennsylvania.)

In the Second Geological Survey of Pennsylvania, these three formations are considered under one heading. The uppermost layer or white Medina is made up chiefly of massive beds of hard white and greenish-gray flinty sandstone; fine grained, compact, homogeneous, and in most parts of these two counties contains few pebbles.

The lower division or red Medina consists of an upper portion of red sand rocks and a lower portion of alternate layers of sandstones and shales. The Oneida consists of gray ochre pitted sandstones, the lower strata of which are quite hard.

The combined thickness of the Medina and Oneida frequently varies from 1000 to 2000 feet. The upper or white Medina usually consists of approximately half of the total. The other two are frequently of equal thickness but vary greatly.

In some places these beds lie nearly horizontal and at others are inclined at various angles. The hard white Medina forms the tops of the ridges. Lower down, the red Medina, which is softer, has weathered away and still lower, the Oneida, which is harder than the red Medina but softer than the white, projects and forms terraces along the sides of the hills. As the red Medina weathers away the hard white material breaks off and forms extensive ganister floes ranging from a few to many feet in thickness, such as are shown in Figs. 6 to 10, inclusive. This talus rock is the material usually used in the manufacture of silica brick, but in some cases it is quarried from the solid formation as is shown in Fig. 11. In the neighborhood of this quarry the white and red formations have approximately the following composition:

	White	Red		White	Red
	Per cent	Per cent		Per cent	Per cent
Loss on ignition	0.15	0.42	MgO	0.02	0.03
SiO_2	99.17	97.77	TiO_2	.02	.07
Fe_2O_3	.06	.36	Alkali	.13	.21
Al_2O_3	.45	1.20			
CaO	.02	.02		100.02	100.08

A quartzite which is quarried in Lehigh County, Pa., for silica brick purposes is also probably white Medina.

Small percentages of the red Medina are sometimes used in conjunction with the white, and in some places some of the purer portions of the Oneida formation are used. The glass sand quarry shown in the foreground of Fig. 10 is in Oriskany quartzite. For

FIG. 6.—*Medina ganister floe, Juniata River, near Mount Union, Huntingdon County, Pa.*

FIG. 7.—*Medina ganister floe, north side of Juniata River, near Mount Union, Huntingdon County, Pa.*

PLATE 8. Outcrop of Medina top of Jack's Mountain, Huntingdon County, Pa.

FIG. 9.—*Ganister talus slope on Jack's Mountain, formed from Medina outcrop [illegible] Mapleton, Huntingdon County, Pa.*

FIG. 10.—*Medina ridge in background, showing talus slope. Pennsylvania Glass Sand Co. Quarry (Oriskany formation) in foreground. Looking northeast along strike, Mapleton, Huntingdon County, Pa.*

Fig. 11. Medina quartzite showing dip of strata, Brooks Mill, Elk County, Pa.

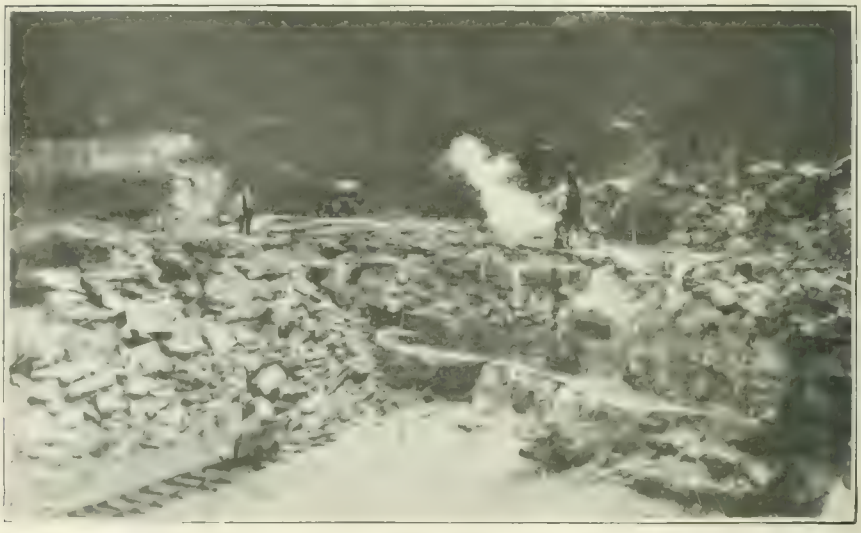


Fig. 12. Horizontal quartzite quarry, line of separation between upper and lower [illegible] horizontal and 3 feet below drill set up, Layton, Fayette County, Pa.

glass sand purposes the loose grained weathered material from strata which are on edge is much sought after. However, there are localities where the Oriskany is a hard, firmly bonded quartzite of good purity. In such places it should be suitable for the manufacture of silica brick.

In Fayette County, Pa., the homewood sandstone formation of the Pottsville series is used to some extent. It has not been metamorphosed quite as much as most quartzites which are used for silica refractories, and hence is slightly softer. It is usually quarried as shown in Fig. 12. It usually occurs in two well defined layers. The upper is white and large grained, and at times has been used as glass sand. The lower one is finer grained, gray and less pure. In the view the line of separation between the two is approximately 3 feet below the drill set up. The composition of the two strata in the neighborhood of this quarry are:

	Upper	Lower		Upper	Lower
	Per cent	Per cent		Per cent	Per cent
Loss on ignition	0.30	0.50	MgO	0.03	0.05
SiO_2	98.82	97.44	TiO_2	.03	.13
Fe_2O_3	.06	.17	Alkali	.11	.19
Al_2O_3	.60	1.57			
CaO	.02	.02		99.97	100.07

In the manufacture of silica brick these two materials are frequently mixed together in equal proportions.

Wisconsin probably ranks second in the production of quartzite for silica refractories. A typical analysis of this material is as follows:[20]

	Per cent
SiO_2	97.15
Al_2O_3	1.00
Fe_2O_3	1.05
CaO	.10
MgO	.25
Alkali	.10
	99.65

Here the rock all comes from the Baraboo formation of the Devils Lake region. This formation which is a true sedimentary quartzite, consists of a series of bluffs or ranges several miles long and wide and several hundred feet high. The upper portion which forms the great bulk of the formation and is the part used for silica refractories, consists of medium-sized, rounded grains

[20] K. Seaver, Am. Inst. Min. Engrs., Trans., 53, pp. 127–128.

of quartz cemented together by secondary deposition of quartz, forming a tightly interlocking mass. Its color is generally pink or shades off into purple or gray. Only in a few places is it white.

Scattered through the massive quartzite are layers of quartz schist varying in thickness from a few inches to several feet. These are parallel to the bedding planes and have been found to have originally contained slightly more clay than the rest of the material. At the bottom of the formation the quartzite gives way to a highly siliceous conglomerate. Quarrying has been conducted chiefly at two places—Devils Lake, which is located on what is known as the South Range, and at Ablemans, which is on the North Range. Crushers are usually located near the foot of the bluffs. Hence detached fragments scattered over the hillsides may be used or the material may be blasted off the solid. In locating quarries in this region care is usually taken to see that the rock contains a sufficiently high percentage of silica, and that the quarry site is free from certain strata, which occur throughout these deposits and which are considered objectionable. A considerable part of the material used from this region thus far for silica brick has consisted of boulders collected from the surface.

The two following districts are described to give an idea of the variety of formation from which rock for silica refractories may be obtained.

In the region covered by the Pueblo (Colo.) Folio of the United States Geological Survey, the Dakota formation, from which rock for silica refractories is taken, is practically all quartz sandstones interlaid with thin shale beds. Its total thickness is usually several hundred feet. The beds vary greatly from place to place and tend to be more porous in the lower portion than near the top. In this district the Dakota lies unconformably upon Juratrias beds, thus omitting the Commanchean. At the top the Dakota passes gradually into shales of the next cretaceous formation without any sharp line of separation. Toward the top the shaly members of the Dakota become gradually more numerous and the sandy members thinner until the latter cease altogether. The highest sandstone is usually a single layer of dense, brittle rock having a vertical fracture. In these upper layers shales suitable for fire clay purposes occur. The sandstones are usually light gray, pink or white, weathering at the surface to yellow, orange, and brown. They vary considerably in friability; therefore it is necessary to exercise care and select only firmly bonded materials.

In Montana material for silica refractories is obtained from the Quadrant formation which is described as follows in the Philipsburg (Mont.) Folio of the United States Geological Survey. In this district the Quadrant formation is the only representative of the Pennsylvanian series. It rests unconformably upon the Mississippian series and is overlain unconformably by the Jurassic. It consists of two members. The lower one is a series of red magnesian limestones and shales. The upper member, mainly quartzite, comprises three strata, the lowest consisting of light-colored, pure, thick-bedded quartzite; the middle of calcareous shale and impure cherty limestone; and the uppermost of somewhat impure quartzite and quartzitic sandstone. The lower massive quartzite is apparently 350 feet thick in places, and is probably the more valuable rock for silica refractories because of its purity and dense structure, although the upper beds are also of considerable thickness and may furnish suitable material at certain places.

The material used in the manufacture of silica brick in Alabama is a hard quartzite with tightly interlocking grains. It occurs as loose boulders which are present in large quantities and appear to be from one of the older formations, probably the Weisner formation of Lower Cambrian age.

The geologic ages of the various materials used for the manufacture of silica brick in the United States are shown in part in Table 5.

TABLE 5.—Geologic Column with Reference to Commercial Quartzites for Silica Refractories

Major divisions				Materials used commercially
CENOZOIC ERA				
Quaternary	Pleistocene.			
Tertiary	Pliocene.			
	Miocene.			
	Oligocene.			
	Eocene.			
MESOZOIC ERA				
Cretaceous	Laramie.			
	Montana..	Western Interior.		
	Colorado..			
	Dakota....			
Comanchian	Washita....		From these come...	Colorado quartzites.
	Fredericksburg..	Western Gulf Region..		
	Trinity.....			
Jurassic.				
Triassic.				

TABLE 5.—Geologic Column with Reference to Commercial Quartzites for Silica Refractories—Continued

Major divisions				Materials used commercially
PALEOZOIC ERA				
Permian	Dunkard.			
Pennsylvanian (Upper Carboniferous)	Coal Measures	Monongahela	Upper productive.	
		Conemaugh	Lower barren.	
		Allegheny	Lower productive.	
	Pottsville (Millstone grit)	Homewood sandstone	Quadrant Formation of Western Montana	Homewood sandstone, Pennsylvania. Montana quartzite.
		Mercer shale, coal, and fire clay		
		Connoquenessing		
		Sharon shale and coal		
		Sharon or Olean conglomerate.		
Mississippian (Subcarboniferous)	Kaskaska (Chester)			
	St. Louis	Mississippi River States	Mitchell Limestone Formation	Indiana chert.
	Osage (Augusta)			
	Kinderhook			
Devonian	Upper	Chautauquan (Chemung–Catskill groups).		
		Senecan	Portage–Naples–Ithaca groups.	
			Genesee–Tully groups.	
	Middle	Erian	Hamilton group.	
			Onondaga group.	
	Lower	Oriskanian (Esopus–Oriskany groups).		
		Helderbergian (Becraft – New Scotland – Coeymans Formations).		
Silurian	Upper (Cayugan)	Monroe group. (Manlius the youngest formation.)		
		Salina group.		
	Middle (Niagaran)	Guelph dolomite	Louisville limestone.	
		Lockport dolomite		
		Rochester shale		
		Clinton Formation.		
	Lower (Oswegan)	Medina	White Medina	Medina quartzite, Pennsylvania.
			Red Medina	
		Oneida		Oneida quartzite, Pennsylvania.
Ordovician	Upper (Cincinnatian)	Gamache.		
		Richmond.		
		Maysville and Lorraine.		
		Eden and Utica.		
	Middle (Champlainian)	Collingwood.		
		Trenton.		
		Black River.		
		Lowville.		
		Chazy and Stones River, St. Peter.		
	Lower (Canadian)	Fort Cassin.		
		Beekmantown.		
Cambrian	Upper or Potsdam.			
	Middle or Arcadian.			
	Lower or Georgian. (Probably Weisner Formation)			Alabama quartzite.
PROTEROZOIC (ALGONKIAN)				
Keweenawan.				
Huronian	Upper.			
	Middle (Baraboo formation, probably Middle Huronian)			Wisconsin quartzite.
	Lower.			
ARCHEOZOIC (ARCHAEAN)				

An inspection of this table indicates what a great variation there is in the various materials in this respect.

3. MANUFACTURE

The following remarks describe the general method of manufacture at a plant which uses Pennsylvania Medina quartzite

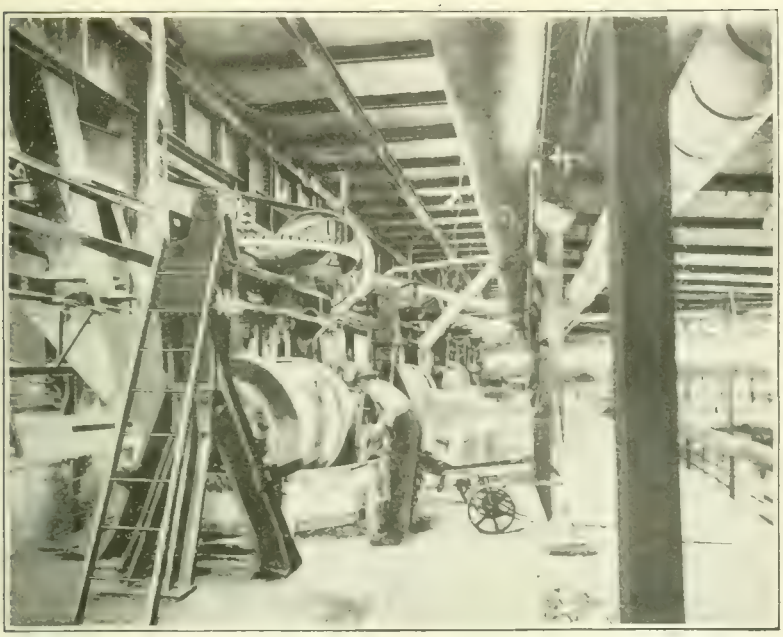


FIG. 13.—*[illegible]* (*Harbison & Walker plant*)

FIG. 14.—*[illegible]* *on dryer cars* (*Harbison & Walker plant*)

FIG. 15.—*Kilns for burning silica brick, showing holes in crowns of kilns for cooling, and ganister floes in distance (Harbison & Walker plant)*

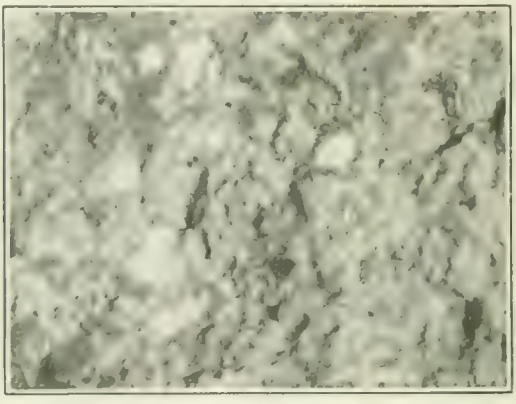


FIG. 22.—*Brick made from Modena quartzite, after* [illegible] *to 1450° C.; groundmass is tridymite, gray outer portion of large particles is tridymite in* [illegible] *white centers of large particles are cristobalite (natural size)*

Most other silica brick plants in the United States follow approximately this procedure except as noted below.

The rock direct from the quarry is crushed to pass through a 2-inch ring. Washing of the rock to remove foreign or earthy matter may be resorted to at certain places. The material is then taken in a car to the wet pan and there dumped. Each charge for a pan weighs about 1250 pounds. A little water is added and the material is ground for 15 minutes. After 10 minutes of this grinding, 2 per cent of lime by weight is added as slaked high-calcium lime, in the form of a thin slip. It is run through a spout to the middle of the crushing surface of the pan, while the pan is in motion. This is well shown in Fig. 13. In this way the lime is thoroughly mixed with the quartzite. When the grinding has continued for the full 15 minutes the mix is ready for molding and is removed from the pan to a tramcar by means of a mechanical scraper.

The lime is prepared by weighing out enough quicklime to furnish exactly 2 per cent of CaO to the weight of quartzite. The weighing out is done on the basis of an analysis. Thus, if the lime contains only 90 per cent CaO, proportionately more is added. This lime is then placed in a small mixer or blunger; is slaked and stirred up to the proper consistency; and then run through a screen of approximately 10 meshes per linear inch on its way to the wet pan. Hydrated lime is sometimes weighed into the mixer instead of quicklime and appears to give exactly the same results.

From the pan the mix is carried to an elevator, raised to an upper floor, and dropped into a container, from which the workmen use it directly in filling the brick molds. This container is in the nature of a cylinder approximately 5 feet in diameter and 10 to 15 feet tall, with slatted openings at the bottom, so that the material can be removed readily. The slats can also be removed. The molders work at benches adjoining the outlets of the containers. The brick molds are of iron and consist of a bottom plate on which lies a 3-brick form. The brand on the brick is formed by the bottom plate of the mold. Directly above the molds is situated a screen which can be shaken by the operator. Pennsylvania washed glass sand is placed upon this screen and a slight jar is sufficient to shake down enough sand to sand a set of molds.

As shown in Fig. 14, a mass of mix is wadded together with the hands and is then thrown heavily into the molds and rammed in,

the surplus struck off and the bricks turned out onto an iron pallet. These pallets are then placed on drier cars. When a car is full it is left standing in the open for a few minutes and is then run into a hot-air drier. Regular burn brick are made up just wet enough so that they will fill the corners of the molds readily. The mix for large shapes is made slightly drier than for regular brick, but the grind is longer, approximately two or three pans per hour instead of four. (The grind for regular brick is at the rate of four pans per hour.) This extra grinding aids in producing a more plastic consistency. The material for large shapes is pounded into the molds with heavy wooden mallets. As the jars due to the plant engines and passing trains tend to cause large shapes to deform before they get their initial set, the stiffer they are when they come from the mold the better.

Of course the time of grinding at each plant will depend somewhat upon the nature of the quartzite used and the ware being turned out. Some plants make up brick as described above, and when the bricks are partially dry they are re-pressed. Another way is to have the mix somewhat drier to start with and make the brick up directly on a dry press machine.

Practically all types of driers used for drying heavy clay wares have also been applied to drying silica wares. Perhaps the most satisfactory type from an economical standpoint is a Richardson type tunnel drier, using waste heat from cooling kilns, and if necessary an auxiliary heater, either a small furnace and brick checkerwork, or else by passing air over exhaust steam pipes. Silica brick give practically no trouble in drying. The only requirement is that the brick must be bone dry before being set in the kilns. Otherwise, they break under the pressure when set, or tend to slump and break during water smoking.

As shown in Fig. 15, the kilns are of the regulation circular down-draft type, varying from 30 to 40 feet in diameter. The bag walls are usually low, as direct fire does not appear to affect the brick much. Regular 9-inch silica brick are set approximately 3 to 3½ on 1 or, in other words, ½ inch is left between the bricks to allow for expansion. They are put in benches 4 feet wide by the width of the kiln by the height of the kiln and a space of 4 inches is left between benches, and approximately 18 inches between the top of the bench and the crown of the kiln. This latter is done to prevent lifting of the crown of the kiln by the expanding brick. Thus, the benches are from 12 to 15 feet high. As set in the kilns there is a pressure of approximately 15 pounds

per square inch on the bottom brick, which do not crush or crack if they are thoroughly dry. A course of bats is put in occasionally to level up the bench horizontally, but they are not used in any way to take the load off the unburned brick. In general the time of burning is nine days with a maximum heat treatment equivalent to cone 14–18. Five to eight additional days are required for cooling.

A departure from the usual method of burning, which is used by some plants, is the continuous-tunnel kiln. With these kilns the period of burning is approximately a week, and the cooling takes another week. The maximum heat treatment is equivalent to cone 12–14.

In England and other European countries brick which contain 96 per cent or more of SiO_2 are manufactured in a manner similar to that used for making silica brick in the United States, except that the methods of mixing, etc., vary somewhat. It is a frequent custom in England to use a somewhat lower percentage of lime (1.3 per cent or even less) and add a small percentage of a highly siliceous clay to strengthen the brick in the raw state. If clay were not added to these brick, they would be weak because of their low lime content.

In England segmental gas retorts are frequently made from materials which contain 85 to 92 per cent of SiO_2. Such pieces are, of course, made by mixing larger percentages of siliceous clays with the silica, and hence, do not correspond to silica brick as manufactured in the United States.

4. USES IN INDUSTRIES

Silica refractories are particularly useful in industrial work. This is due to their rigidity at temperatures above the range of usefulness of ordinary fire-clay brick, and to the fact that they expand slightly when heated instead of shrinking as clay brick do.

For by-product coke-oven work the brick in the regenerative and lower two-thirds of the coking chamber should have a high fusion point, as an extreme temperature of 1510 to 1540° C is sometimes reached. The brick in the lower two-thirds of the coking chamber, where they come in contact with the coal and coke during charging and unloading must also resist abrasion. The upper one-third of the coking chamber may be made of lower fusion point material and need not be particularly resistant to abrasion, as here the temperature rarely exceeds 1320° C and the brick are seldom touched by coal or coke. Occasionally charges of coal have been

found to expand in the coking chambers, so that when coked they could not be removed by the usual mechanical device. Trouble from cracking and spalling may sometimes occur if the ovens are heated up too rapidly. However, when the temperature of a series of these ovens is once raised to the operating temperature, they should last for years, except for repairs occasioned by abrasion, etc.

From the work of Blasberg [21] we gather that silica refractories which have been used in Siemen's by-product coke ovens frequently show an increase in iron and always a considerable increase in alkalies, particularly the brick used in the checkerwork. In many cases they contain up to 1 per cent of carbon. It is assumed that the carbon comes from the dissociation of gaseous hydrocarbons or carbon monoxide, and that the other contamination comes from the coal ash. The conclusions drawn are that for this work silica brick should be as low in porosity as possible and that the gases should be free from flue dust.

In open-hearth steel furnace work, silica brick are used for the top, ends, and sometimes for the side walls of the furnace. Here the chief essentials are that the brick have a high crushing strength when hot and high resistance to spalling and slagging, as well as high softening temperature.

The port bulkheads at the ends of these furnaces are subjected to a severe sand-blast action from particles of limestone and iron oxide, which are blown over from the hearth of the furnace. As these materials both act as active fluxes on silica brick under reducing conditions, the result is that these bulkheads are rapidly eaten away by a combined cutting and slagging action. The 13-inch brick used in these bulkheads wear away so fast that it is frequently necessary to replace them in two or three weeks. Crowns of such furnaces are also somewhat attacked by the slagging action of lime and iron oxide which are blown up against them and also by the dust of the gaseous fuel used. Thus, an average crown lasts approximately 300 heats when natural gas, which is quite free from dust, is used as a fuel; approximately 200 heats with producer gas, which contains considerable dust, and somewhat less than 200 heats when powdered coal is used.

For steel furnace work it is desirable that the softening temperature (as described below) of the brick be somewhat above that of Orton cone No. 31. Even then, at times the surface of the bricks

[21] Stahl, W. Eisen, **30**, pp. 1055–1060; 1910.

will melt and hang down in long white stalactite like forms from the crown of a furnace.

The uses of silica refractories in copper reverberatory furnaces are similar to those in open-hearth steel furnaces, except that for melts of lead-free metallic copper, silica can be used throughout the furnace except at the slag line. For this work the brick should be as low in porosity as possible to avoid absorption of copper oxide, which slags away the bricks when the furnace is at its maximum temperature. Silica brick arches in copper furnace work wear much better if they are not insulated. The maximum temperature reached in the copper refining furnaces is approximately 1350° C; hence, for this purpose some refractoriness might be sacrificed if anything is to be gained thereby in decreased porosity and resistance to spalling.

In glass furnace work silica refractories are used for furnace roofs, and are useful for stiffening up walls, etc., made of clay brick, which would otherwise soften and sag from the effect of heat and fumes arising from the glass.

IV. STATEMENT OF WORK DONE AND DESCRIPTION OF METHODS

1. GENERAL STATEMENT

The experimental work consisted of a number of heating tests of silica materials carried out for the purpose of determining the amount, speed, and nature of the volume changes occasioned in such materials by various heat treatments. The work includes the study of raw quartzites, raw commercial brick mixes, commercial brick, and special mixes. In addition to this the properties of finished brick and some data concerning the manufacture and qualities of materials have been considered. Wherever possible one number has been used to represent the quartzite, the burned and the unburned brick of a variety, the distinction in each case being made in the text. This applies to Tables 6, 7, 8, 12, 14, 16, 17, 18, 19, 20, and 21.

2. HEATING TESTS

(*a*) *Series 1.*—The first work carried out was to determine the exterior volume changes of a silica brick mixture on repeated burnings at successively higher temperatures. The material (No. 6) used in this first test consisted of twelve 2-inch cubes made from Medina quartzite, plus 2 per cent of lime. It was

ground and mixed in the usual way at a silica brick plant. Its screen analysis should correspond approximately to that given for regular grind (Table 2). When the cubes had been thoroughly dried at 110° C they were burned to 1150° C in approximately three days before any measurements were made upon them. After this burning the exterior volumes of the pieces were taken by means of a Seger voluminometer, using a petroleum oil as the displacing liquid. By means of a micrometer caliper, one dimension of each piece was also measured. The pieces were then burned again, this time to 1200° C, after which the volume, etc., of each piece were again measured. The next burning temperature was 1250° C. These burnings and measurements were continued at 50° C intervals up to and including 1500° C. The pieces were burned three times to 1500° C. The duration of these burns varied somewhat but the average was as follows:

Eighteen hours for heating to 800° C and six hours for heating from 800° C to the desired temperature. The pieces were held at the maximum temperature for one and one-half hours in each case. In all succeeding heating tests throughout this paper this heating procedure was followed except as noted. The volume and linear expansions were calculated in terms of the first set of measurements (those made following the 1150° C burn). Starting with the 1200° C burn one piece was saved from each burn up to and including the 1500° C burn for microanalyses.

(*b*) *Series 2.*—The above work showed such interesting results that it was decided to extend it to other varieties of material. This second test included raw quartzite, raw brick mixes and commercially burned brick. The burnings were made as with the preceding set and ranged from 1200° C to 1500° C, inclusive. The materials used in this case were two 2-inch cubes each of Medina (No. 6), Baraboo (No. 22), and Alabama (No. 26) quartzites, 12 cubes of Medina raw brick mix as used for the first test, 12 cubes of a commercial brick made from Baraboo quartzite (No. 18) and 12 cubes of a brick made from Baraboo quartzite which had been lightly calcined (No. 20) before being made into brick.

In this case the wet weight, dry weight, and suspended weight of the pieces were taken, in addition to the measurements made in the first test. These latter determinations were made by drying the pieces at 110° C, weighing dry, saturating with water and weighing wet, then weighing suspended in water which was at room temperature. Saturation was obtained by placing the pieces in an iron cylinder, covering them with boiling water, and subject-

ing them, while thus submerged, to a vacuum equivalent to 24 inches of mercury for a period of four hours. In the case of the raw brick mix it was necessary to make these weights in petroleum of a known specific gravity, for water would slake the material. The wet, dry, and suspended weight method of determining porosity, etc., was used throughout these tests, except as noted.

(*c*) *Series 3.*—The above studies indicated the desirability of knowing the effect produced in quartzite and silica brick mixes at different stages during a single burn. Hence a burn was conducted from which pieces were withdrawn from time to time. In this case the material used consisted of 42 pieces (approximately 50 g each) of Baraboo (No. 18) quartzite, and 42 two-inch cubes of a mix made by mixing 2 per cent of lime with this Baraboo quartzite that had been crushed to pass through an 8-mesh screen. The volumes, etc., of the pieces were taken, after which they were placed in a kiln in 42 small numbered clay saggers, each sagger containing one piece of quartzite, one cube, and the desired Orton cones. These saggers were covered and set two high, according to number. Heating to 1000° C took approximately two days, at which temperature box number one was withdrawn. The temperature was then raised to 1200° C and held at this temperature for one hour to insure uniformity of kiln temperature, at the end of which time box number two was withdrawn. Four more draws were made at this temperature at intervals of five hours, after which the temperature was raised to 1250° C in two hours, held for one hour, and the first 1250° C draw made. This process was continued by 50° C steps up to 1450° C, at which temperature nine draws were made instead of five, before proceeding to 1500° C, which was the highest temperature used. To prevent spalling of the materials when withdrawn from the kiln, they were cooled slowly by being placed in successively cooler kilns.

(*d*) *Series 4.*—At this juncture it was apparent that the changes taking place in commercial brick when heated once to a temperature of from 1400 to 1500° C in a manner similar to that used in series 1 and 2, were a guide to the degree of burning which the brick had received in manufacture. Following this line, several of the leading varieties of silica brick manufactured in the United States, and the corresponding quartzites and raw mixes, were tested by single heatings at temperatures of 1400, 1450, and 1500° C. Some of these heatings were carried out at the same time as those of series 2.

(c) *Series 5.*—Working with so many different varieties of material it was soon seen that various constituents occasionally present or added and treatments such as calcining of the quartzite before making into brick, had certain effects on the burning behavior of the wares. To determine these effects, several heats were made. Tests were carried out with iron oxide, impure ferruginous clay, pure plastic clay, sodium chloride, lime, and calcium fluoride. Loose-grained Medina (No. 37) quartzite crushed to pass through an 8-mesh screen was used in this set of experiments except as noted.

The first work consisted of two similar burns on the same pieces. The maximum temperature in each of these burns was 1400° C and the heating was as follows:

Fifty degrees C in 15 minutes from atmospheric temperature to 500° C, 75° C in 15 minutes from 500 to 800° C, 100° C in 15 minutes from 800 to 1200° C, then 50° C in 15 minutes to 1400° C and held at this maximum temperature for one and one-half hours. In this case only exterior volumes were measured. Exterior volume changes alone do not constitute a very definite indicator of the amount of transformation taking place, while as shown later, the specific gravity change in conjunction with the exterior volume and porosity changes do constitute a definite indicator of the nature of the changes taking place, and the specific gravity alone indicates the exact degree of transformation. Hence, in the balance of the work in connection with added ingredients specific gravity was used as an indicator of the degree to which the materials had been transformed.

The second test was conducted with the 1450° C burn of series 4. Hence, the figures of the present test are more or less comparable with those of the former. The third test was conducted to determine the effect of coarser grind, pressure in molding, and a slightly longer time of burning. In this case the quartzite was broken up in a jaw crusher to pass through a 4-mesh screen, and the mix was made up by hand to avoid further crushing of the material. The test pieces were molded under a pressure of 5000 pounds per square inch. In burning the temperature was raised to 800° C in approximately 18 hours, then to 1200 ° C in 4 hours, and was maintained between 1200° C and 1300° C for 2¾ days. Finally, the temperature was gradually raised to 1450° C and held at this maximum for 3 hours. A fourth test of added ingredients was conducted in connection with series 7.

For the tests of calcined materials, Baraboo (No. 18), Medina (No. 6), and loose-grained Medina (No. 37) quartzites were used. The materials in lump form were calcined twice to 1500° C, then crushed to pass through an 8-mesh sieve before being made up in a mix. The first of these tests was carried out in conjunction with test No. 2 above (this series) and the second test (the pieces for which were made up with a pressure of 5000 pounds per square inch) was in conjunction with the second burn of test No. 1 above (this series). Another test of these materials was made in conjunction with series 7.

(*f*) *Series 6.*—During the time that the above tests were being made, a number of tests of quartzites to determine their suitability for the manufacture of silica brick, were also being carried out. The first material to be tried out was the friable Medina (No. 37) described under series 5 above.

In these tests the fineness of grind, added ingredients, and method of burning were varied. The first test consisted of two mixes, one of 8-mesh material and one of 10-mesh material, each of which contained 2 per cent of CaO. Brick from these mixes were burned as in tests of series 1. The maximum temperature (1465° C) was maintained for only a few minutes. This treatment brought down cone 16. The second test was of material (No. 37) which had been previously calcined at 1300° C for 1½ hours and then quenched with water, and crushed to pass through an 8-mesh sieve. This mix was made up with 2 per cent of lime and burned the same as above, except that in this case a maximum temperature of 1485° C was maintained for a few minutes, which brought cone 17 down. The third burn consisted of mixes made up from 8-mesh material (No. 37) and 2.5 per cent lime, 3 per cent of lime, 1 per cent lime, plus 1.5 per cent Tennessee ball clay, and 2.5 per cent of Tennessee ball clay, respectively. These brick were burned same as the above to cone 16+ with a maximum temperature of 1485° C which was maintained for a few minutes. The fourth burn was for the purpose of reburning previously burned bricks and consisted of two bricks each of the mixes containing 2.5 per cent of lime, 3 per cent lime, and 2.5 per cent Tennessee ball clay, respectively, and the mix made from calcined quartzite plus 2 per cent lime. In this case the burn was similar to the preceding, except that the maximum temperature of 1450° C was maintained for five hours. The fifth test consisted of bricks made from 8 and 20 mesh materials (No. 37), respectively. In

each case 2.5 per cent of lime was used in the mix. The fifth burn also contained brick made from a strongly bonded quartzite (No. 39) having a conchoidal fracture. This rock was crushed to pass through an 8-mesh screen, and 2.5 per cent of lime was used in the mix. This burn was conducted similarly to the others, and the maximum temperature (1470° C) was maintained for 3 hours. This brought down cone 20. Brick made from commercially used Medina quartzite was tested in conjunction with these various sets of bricks.

(*g*) *Series 7.*—Satisfactory data of the relation existing between results obtained in test kilns and those obtained in regular silica brick kilns were lacking; hence, a burn was made in a commercial silica brick kiln. The materials of this test consisted of one piece of the preburned Baraboo mix described under series 5 above—shale and lime mix, sodium chloride lime mix, and a piece which contained only lime in addition to the quartzite. The materials were placed near the top of the kiln. The period of burning was 14 days, and the period of cooling was seven days. The heat treatment received by the pieces brought down cone 16+.

(*h*) *Series 8.*—This test was conducted to determine the comparative behavior of underburned, medium burned, and well-burned bricks on being rapidly reheated. The brick were made from Medina (No. 6) quartzite, and all came from the same kiln of ware. During the reheating test the temperature was raised to 1480° C in approximately six hours, and this maximum was maintained for approximately one hour.

3. PROPERTIES OF FINISHED BRICK AND DATA ON MANUFACTURE

(*a*) *Data on Commercial Bricks.*—The foregoing tests brought out the fact that the specific gravity of silica brick, as they come from the manufacturer, calculated from the wet, dry, and suspended weights, is the best guide in showing the degree of burning which the various brands receive in manufacture. Likewise, it was seen that brick made from any one variety of quartzite show certain peculiarities. It was learned from the different manufacturers that the time of burning and the cone representing the maximum of the heat treatment given in each case varied considerably. To determine the specific gravity of average burned brick the specific gravities of most of the leading varieties manufactured in the United States were taken, and to show the prop-

erties of the different quartzites these data have been arranged according to the quartzite from which the bricks were made.

(*b*) *Analyses and Softening Temperatures.*—The softening temperature of a material may be the limiting factor in its use. Hence, this point has been considered for a few special materials, and since the chemical composition of silica brick is so closely allied with the softening temperature, these two subjects are considered together.

(*c*) *Load Tests and Volume Changes at 300° C.*—Load tests have been used to some extent as a guide to the quality of silica refractories. The data here considered have to do chiefly with the rate of heating and a consideration of the volume changes in the neighborhood of 300° C, and in the neighborhood of 1400° C and above. A means of actually measuring volume changes up to 300° C is also considered.

(*d*) *Micro Studies.*—Only such micro studies have been included as are essential to the understanding of the materials used in the experiments. The work taken up includes two Baraboo quartzite draw trial pieces and the corresponding pieces of brick mix, chalcedony cemented quartz conglomerate from Colorado, chalcedony cemented quartz sandstone from Missouri, and chert from Indiana. A general consideration of the optical properties of quartzites which may aid in determining their value for silica refractories is given, as well as a few studies to show the optical properties and nature of the grind of some standard and special silica brick.

(*e*) *Effect of Slags.*—Slag tests in connection with silica refractories have received some attention, and with reference to ware for certain uses are quite important. The work here presented is principally in connection with the effect that fineness of grain has upon slagging of the brick.

(*f*) *Screen analyses.*—Screen analyses have been made of 15 representative raw brick mixes. Water was used to disintegrate the material and for washing.

(*g*) *Cold Strength Tests.*—Cross breaking tests were made of 18 varieties of commercial brick. Standard 9-inch brick were used. The brick were placed on edge on knife edges, with 6 inches between supports, as described elsewhere under cold cross breaking test.

V. RESULTS OF WORK AND DISCUSSION OF SAME

1. HEATING TESTS

(*a*) *Series 1.*—Fig. 16 shows the average percentage exterior volume increase of the pieces used for the first test. After the last 1500° C burn the average specific gravity of the five remaining pieces was 2.304. The crystal structure [22] of the cubes after each burn shows that cristobalite was present in appreciable quantities after the 1300° C burn, while tridymite did not make its appearance until the 1450° C burn, after which there was only a trace of it present, although at this stage 81 per cent of the material by volume had been converted to cristobalite. After the first 1500° C burn the tridymite had increased considerably and the amount of crystobalite had somewhat decreased. There was also a slight decrease in the amount of quartz plus silicate glass.

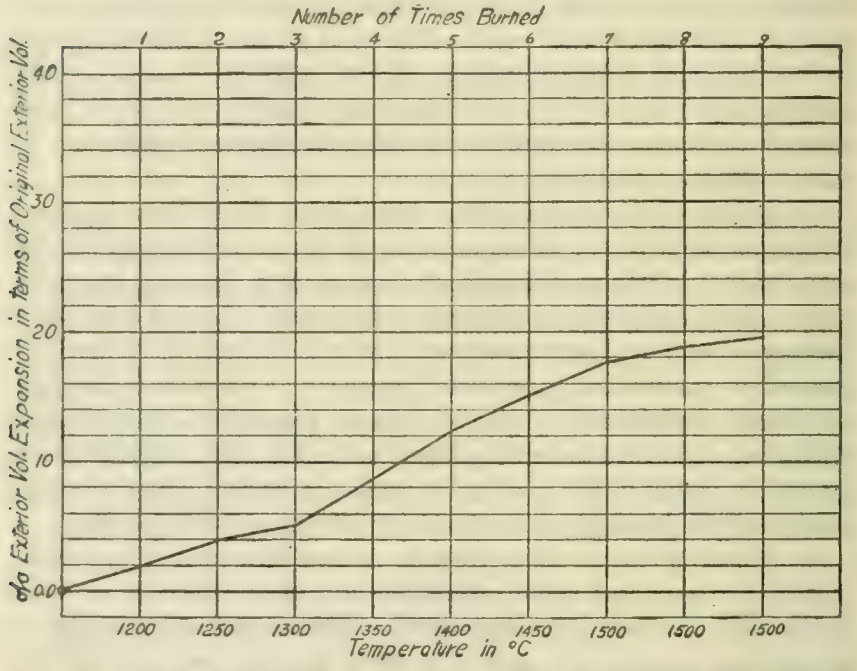


FIG. 16.—*Volume changes caused in Medina mix, which had previously been burned to 1150° C, by repeated burnings to successively higher temperatures from 1200–1500° C*

This series of burns although not directly comparable to a burn in a commercial kiln is nevertheless somewhat analogous, in that to a certain degree the pieces received the effect of soaking during the several burns, and each burn was carried to a higher temperature

[22] Trans. Am. Cer. Soc., 18, p. 519.

than the preceding, thus giving the effect of rising temperature. Such burning differs from commercial burning in the repetition of heating and cooling, and also in the fact that commercial burns are not usually carried to 1500° C. The curve shows the point at which further heating practically ceased to cause additional exterior volume expansion. Hence the specific gravity 2.304 approximately represents the minimum specific gravity obtainable by such heat treatments in which 1500° C is the maximum temperature reached. The crystal changes are of particular interest, in that they show that in silica brick mixes cristobalite and not tridymite is the

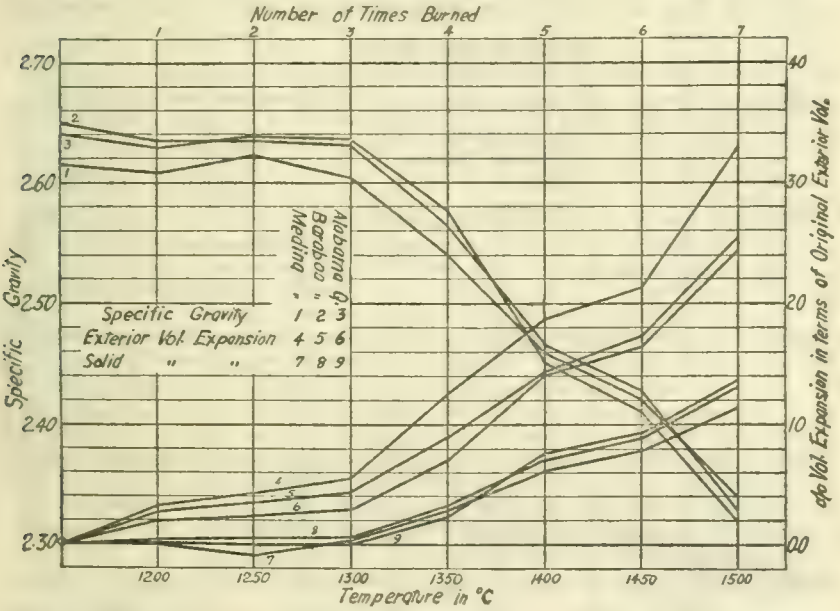


FIG. 17.—*Changes in exterior and solid volumes and specific gravities of Medina, Baraboo, and Alabama quartzites, caused by repeated burnings to successively higher temperatures from 1200–1500° C*

form that appears first and most prominently when such mixes are heated at temperatures below 1470° C. It was found that in these pieces, at temperatures above 1470° C tridymite is still present in appreciable quantities. (After third heating at 1500° C, quartz plus silicate equals 6 per cent, cristobalite 70 per cent, tridymite 24 per cent, by volume.)[23]

(*b*) *Series 2.*—Fig. 17 shows the exterior volume changes and those of the solid portions of the quartzites tested in series No. 2.

[23] Authorities differ as to whether cristobalite or tridymite is the stable form of silica at 1500° C; J. B. Ferguson and H. E. Merwin, Am. J. Sci., 4th series, 46, pp. 417–426.

These changes are figured in terms of the original exterior volumes of the pieces for the sake of convenience in comparison with the other curves. This plan is followed throughout this paper, except as noted. This figure also shows the changes in specific gravity of the pieces.

The specific gravity and solid volume curves show that up to and including 1350° C very little transformation of quartz took place, but that up to this stage there had been some increase in exterior volume of the pieces. By the end of the 1500° C burn, however, almost as much transformation had apparently taken place as would be the case with the corresponding silica brick mixes on

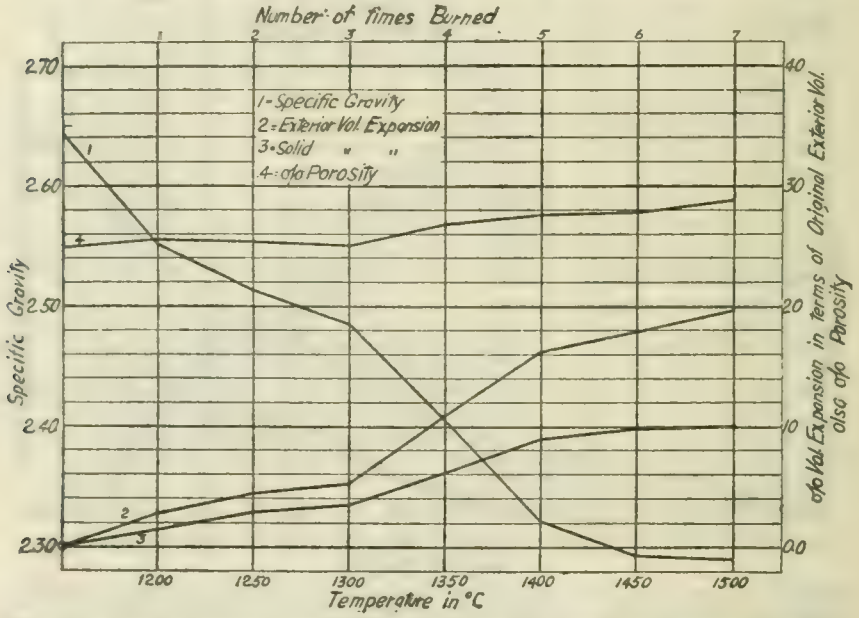


FIG. 18.—*Changes in exterior and solid volumes, specific gravity and porosity, of Medina raw mix, when heated same as quartzites of Fig. 17*

receiving similar heat treatment. It is also seen that the Alabama quartzite, which shows the greatest transformation, as indicated by the curves of specific gravity and the expansion of the solid materials, shows the least exterior volume expansion, while the Medina quartzite which shows the least transformation, shows by far the greatest exterior volume expansion. These three varieties of quartzite, which are fairly representative of the varieties usually used in the manufacture of silica refractories, do not show any great differences in their rates of transformation.

In Fig. 18 are shown the exterior and solid volume changes of the raw Medina mix, as well as its specific-gravity changes. From

consideration of the specific gravity and solid volume expansion curves it is apparent that even at temperatures as low as 1200 and 1250° C there is an appreciable amount of transformation going on in such a mix. An inspection of the exterior and solid volume curves from 1200 to 1350° C shows that there is practically no expansion of pore space during this interval, and possibly a slight contraction. In the region above 1350° C, however, where the rate of transformation increases very rapidly with increase of temperature, it is seen that the exterior volume increase is much more than that of the solid volume, thus showing that the pore space increased considerably. The final specific gravity 2.290, compared to a final specific gravity of 2.304 for the similar material of series 1, shows how close the final specific gravity of two sets of material may be when extensively heated at high temperatures, even though the methods of heating the two sets be somewhat different.

Table 6 shows the exterior volume increase and porosity change for the Baraboo commercial brick, and the brick made of lightly calcined Baraboo quartzite.

TABLE 6.—Effect on Volume and Porosity of Baraboo Quartzite Bricks, Caused by Reburning to Successively Higher Temperatures, in Test Kiln, Same as for Quartzites of Fig. 17

Material	No.	Original per cent porosity	1200° C		1250° C		1300° C		1350° C	
			Per cent volume expansion	Per cent porosity	Per cent volume expansion	Per cent porosity	Per cent volume expansion	Per cent porosity	Per cent volume expansion	Per cent porosity
Brick made from Baraboo quartzite, regular burn	18	22.26	0.190	23.70	0.438	23.36	0.440	23.67	1.731	24.51
Brick made from lightly calcined Baraboo quartzite, burned as above	20	30.09	.108	31.50	.538	31.00	.104	31.38	.412	32.41

Material	No.	Original per cent porosity	1400° C		1450° C		1500° C	
			Per cent volume expansion	Per cent porosity	Per cent volume expansion	Per cent porosity	Per cent volume expansion	Per cent porosity
Brick made from Baraboo quartzite, regular burn	18	22.26	3.408	24.73	4.36	24.80	6.49	25.99
Brick made from lightly calcined Baraboo quartzite, burned as above	20	30.09	.666	31.91	1.04	31.40	1.34	31.93

The brick made from raw quartzite increased considerably in volume and slightly in porosity, while that made from calcined quartzite shows very little increase in volume and with the lower temperatures shows a decrease in porosity. These data make it apparent that commercial brick act essentially as do raw mixes, the chief difference being the lesser amount of transformation which takes place in the commercial brick. Comparison of the data for these two varieties of brick shows what increase in the ultimate degree of burning of a brick a slight calcining of the quartzite will produce.

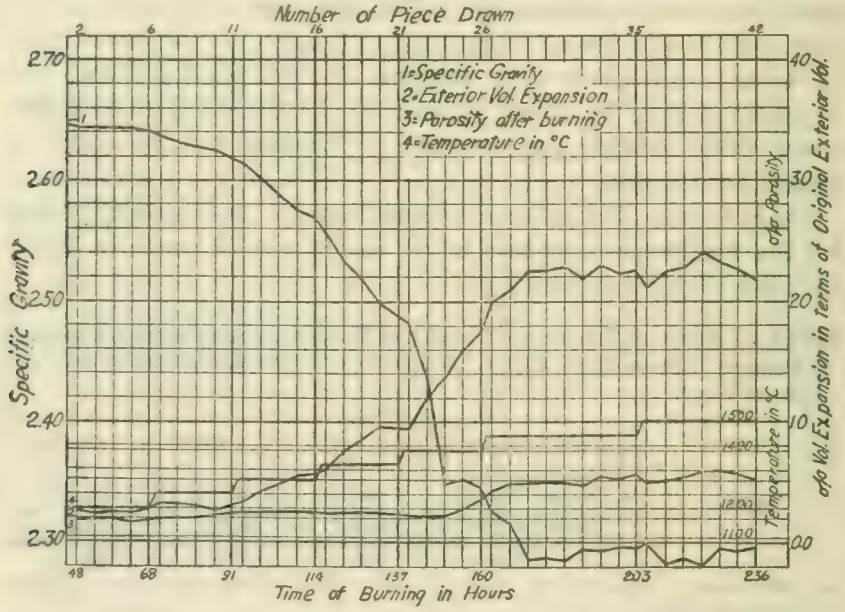


FIG. 19.—*Nature of changes in exterior volume, specific gravity, and porosity of Baraboo quartzite, due to being burned to 1500° C in 236 hours*

Points on the curves were determined by withdrawing pieces at the intervals indicated by vertical lines in the figure.

(*c*) *Series 3.*—Fig. 19 shows the exterior volume, porosity, and specific gravity curves for the 42 pieces of Baraboo quartzite. The original porosity of these pieces is not shown for it was practically zero. This burn, which also included the 42 cubes of Baraboo brick mix, more nearly approached a commercial kiln burn than the heating tests of series Nos. 1 and 2, due to the fact that it eliminated the repeated cooling down and heating up. A comparison of these two methods of burning, however, shows that the repeated burning method approximates a continuous burn fairly well. The duration of this draw trial burn was approximately the same as that of a commercial burn. It differs from a

commercial burn, however, in that a maximum temperature of 1500° C was reached.

The specific gravity curve shows that transformation did not proceed rapidly below 1350° C. This result agrees with that given in series No. 2 for Baraboo quartzite. Also in accordance with series No. 2 transformation took place rapidly above this temperature. The minimum specific gravity was reached considerably before the end of the burn and as shown is approximately 2.290. The exterior volume expansion reached its maximum, approximately 22 per cent, at the same stage at which minimum

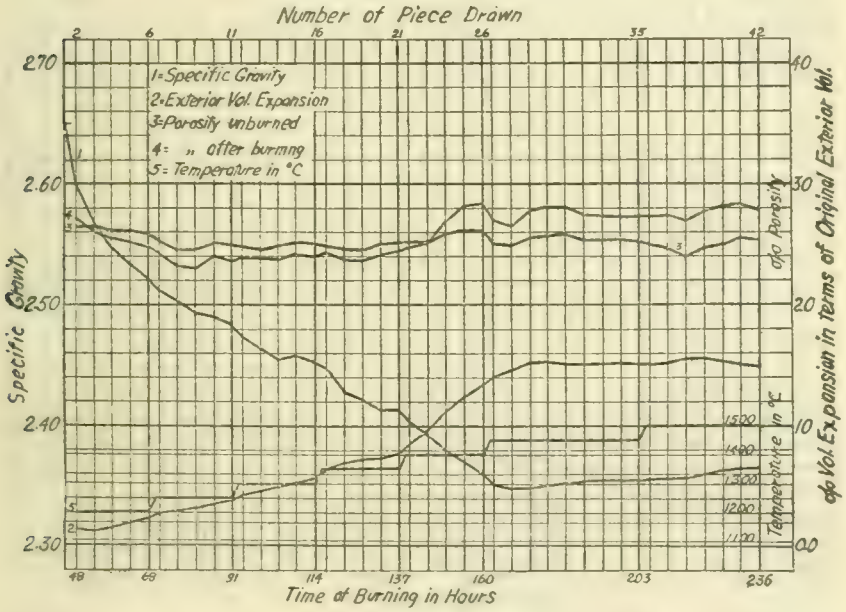


FIG. 20.—*Nature of changes in exterior volume, specific gravity, and porosity of Baraboo mix, due to being burned the same as the Baraboo quartzite of Fig. 19*

specific gravity occurred. The porosity also gradually increased up to this point, after which it remained nearly constant to the end of the burn.

The corresponding curves for the 42 Baraboo brick mix pieces are shown in Figs. 20 and 21. Fig. 21 shows the exterior and solid volume increases for the same pieces as does Fig. 20. In the case of Fig. 20, the original porosity of the pieces is also given.

These curves entirely bear out what has been said concerning the burns of series Nos. 1 and 2. In this case, however, it is plainly seen that the material has reached its maximum exterior volume expansion and its minimum specific gravity. Also in the

part of the burn between 1200 and 1350° C the porosity of the pieces was less than that of the original pieces even though considerable transformation of quartz took place during this interval, as is shown by the drop in specific gravity and increase in solid volume. In fact the expansion of the solid material in the major portion of this region was greater than or equal to the expansion of the exterior volume of the cubes. The maximum depression of the porosity was probably between 1250 and 1300° C. For the higher temperatures, however, the porosity of the burned pieces

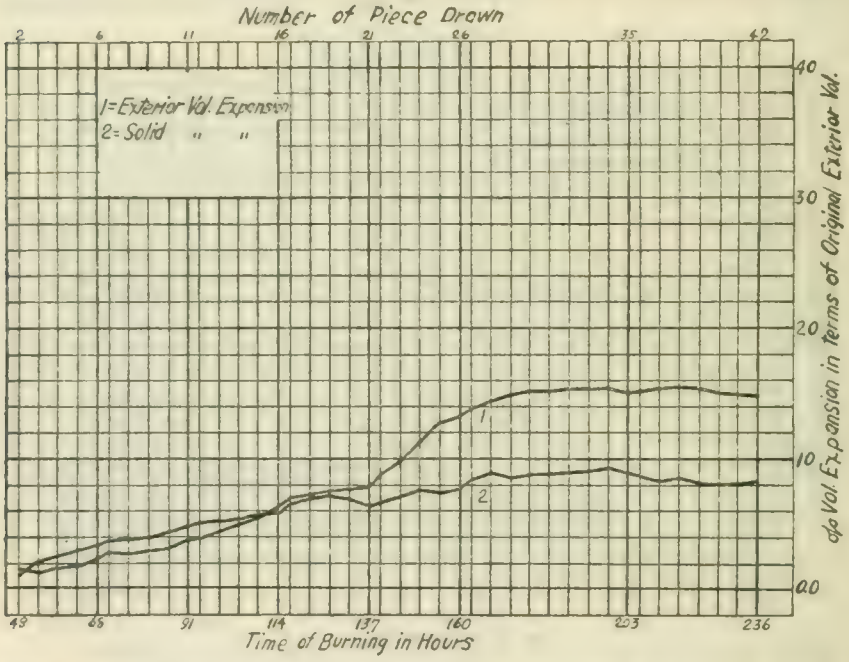


FIG. 21.—*Same materials and conditions as for Fig. 20, showing the relation between exterior and solid volume expansion*

is greater than that of the raw mix. The maximum exterior volume expansion of this brick mix is approximately 15.5 per cent and the minimum specific gravity is in the neighborhood of 2.350, as compared with 22 per cent volume expansion and 2.290 specific gravity for the quartzite pieces.

(d) Series 4.—In Table 7 the results of the single-burn tests of commercial brick are presented. As shown, these include the porosity and specific gravity of the brick as they come from the manufacturer, as well as the porosity, specific gravity, and exterior volume increase of the pieces after burning.

TABLE 7.—Changes in Porosity and Specific Gravity, and Per Cent Exterior Volume Expansion in Terms of Original Exterior Volume, of Commercial Silica Brick, Caused by Single Heatings to Various Temperatures

Number	1400° C				
	Before		After		
	Porosity	Specific gravity	Porosity	Volume expansion	Specific gravity
6	26.46	2.292	26.39	0.64	2.283
4	27.19	2.387	27.60	2.55	2.360
8	31.96	2.342	32.17	1.31	2.323
11	29.56	2.295	29.45	0.42	2.287
18	24.44	2.369	24.93	2.18	2.334
19	25.27	2.434	25.30	2.40	2.387
22	25.37	2.401	25.39	1.62	2.373
20	31.46	2.316	31.63	0.65	2.305
23	22.67	2.496	23.82	6.38	2.389
24	22.67	2.493	23.55	5.62	2.394
25	23.16	2.545	24.68	7.73	2.417
26	30.11	2.316	30.21	0.80	2.309
14	30.64	2.500	31.86	5.22	2.418
27	24.66	2.400	24.66	1.95	2.361
Average, omitting Nos. 23, 24, 25, 17, 30, and 31...	27.93	2.367	28.14	1.794	2.340

Number	1450° C				
	Before		After		
	Porosity	Specific gravity	Porosity	Volume expansion	Specific gravity
6	26.30	2.304	25.53	0.62	2.270
4	27.07	2.412	27.00	3.24	2.333
8	31.67	2.342	31.70	1.01	2.320
11	29.35	2.301	29.45	0.70	2.291
18	23.72	2.393	24.01	1.14	2.378
19	25.00	2.438	25.02	2.20	2.387
22	24.70	2.400	24.77	2.08	2.354
20	31.77	2.338	31.65	0.12	2.326
23	23.77	2.498	24.88	6.61	2.378
24	22.65	2.499	23.42	5.40	2.390
25	23.08	2.537	24.57	7.15	2.415
26	29.39	2.319	28.87	0.37	2.295
14	30.52	2.497	31.80	5.80	2.404
27	24.68	2.402	25.16	2.31	2.366
17	31.35	2.411	32.28	3.51	2.362
30	25.78	2.374	28.40	7.31	2.290
31	24.07	2.392	25.10	2.46	2.365
Average, omitting Nos. 23, 24, 25, 17, 30, and 31...	27.68	2.377	27.70	1.780	2.335

TABLE 7—Continued.

Number	1500° C				
	Before		After		
	Porosity	Specific gravity	Porosity	Volume expansion	Specific gravity
6	26.31	2.277	27.96	2.06	2.280
4	27.21	2.381	33.67	13.05	2.308
8	30.94	2.323	32.78	3.12	2.310
11	30.77	2.278	28.60	−0.13	2.281
18	23.57	2.380	25.67	4.34	2.344
19	25.17	2.417	29.50	11.10	2.334
22	24.29	2.385	28.88	8.88	2.330
20	31.11	2.322	32.06	0.73	2.334
23	23.23	2.490	30.17	17.19	2.333
24	22.67	2.475	31.20	19.70	2.322
25	22.64	2.528	31.41	22.10	2.335
26	29.76	2.298	30.45	0.37	2.306
14	30.59	2.491	38.58	20.91	2.324
27	24.56	2.378	31.55	13.05	2.353
17	30.20	2.393	36.74	13.93	2.317
30	25.67	2.353	30.74	8.80	2.317
31	25.32	2.393	29.93	9.52	2.326
Average, omitting Nos. 23, 24, 25, 17, 30, and 31...	27.65	2.353	30.89	7.040	2.321

Average of determinations on original brick. Average porosity equals 27.75 per cent. Average specific gravity equals 2.366.

These properties are given for the temperatures 1400, 1450, and 1500° C. The average figures given are exclusive of Nos. 23, 24, and 25, which are materials intended for a special purpose, and Nos. 17, 30, and 31, for which the 1400° C heat is missing. The 1400° C heat was maintained for a slightly longer time than the 1450° C heat. This difference, which was first considered negligible, is noticeable both in this table and in connection with the quartzites and raw brick mixes which were also present in these burns. The near approach of the figures of the 1400° C burn to those of the 1450° C burn indicates the effect of this additional heating period.

The average figures given indicate approximately what porosity, volume, and specific gravity changes may be expected from average commercial silica brick with such heat treatments. Hence the magnitude of these changes in commercial brick for such heat treatments is a guide to the degree of burning which the bricks received in manufacture.

TABLE 8.—Changes in Porosity and Specific Gravity, Per Cent Exterior and Solid Volume Expansions in Terms of Original Exterior Volume, and of Exterior and Solid Volume Expansions in Terms of Original Solid Volume, of Quartzites and Raw Commercial Silica Brick Mixes, Caused by Single Heatings to Various Temperatures

Materials	No.	1400 C							
		Before		After					
						In terms of original exterior volume		In terms of original solid volume	
		Specific gravity	Per cent porosity	Specific gravity	Per cent porosity	Per cent exterior volume expansion	Per cent solid volume expansion	Per cent exterior volume expansion	Per cent solid volume expansion
Raw brick mixes:									
Medina	6	2.610	25.25	2.396	26.70	9.31	5.29	12.44	7.06
	4	2.614	26.37	2.409	27.46	8.11	4.63	10.99	6.28
	8	2.609	30.15	2.372	31.56	10.37	5.46	14.80	7.80
Homewood sandstone	14	2.605	28.08	2.407	30.26	9.01	4.05	12.52	5.63
Baraboo	19	2.575	25.23	2.435	26.43	5.82	2.86	7.77	3.81
Quartzites:									
Medina	6	2.615	1.27	2.516	12.61	.....	.....	.....	.....
Baraboo	22	2.650	.00	2.525	3.32	.....	.....	.....	.....
Alabama	26	2.640	.00	2.520	4.04	.....	.....	.....	.....
Homewood sandstone	14	2.628	6.54	2.487	14.68	15.16	4.80	16.21	5.14
Montana	28	2.645	.55	2.497	5.95	11.48	5.40	11.54	5.43
Soft Medina	37	.....	.....	.....	.....	.....	.....	.....	.....

Materials	No.	1450° C							
		Before		After					
						In terms of original exterior volume		In terms of original solid volume	
		Specific gravity	Per cent porosity	Specific gravity	Per cent porosity	Per cent exterior volume expansion	Per cent solid volume expansion	Per cent exterior volume expansion	Per cent solid volume expansion
Raw brick mixes:									
Medina	6	.....	.....	.....	.....	.....	.....	.....	.....
	4	2.631	25.51	2.377	27.49	11.78	6.48	15.78	8.69
	8	2.632	30.40	2.381	32.61	11.47	5.50	16.46	7.90
Homewood sandstone	14	2.617	27.75	2.427	30.32	9.47	4.01	13.11	5.55
Baraboo	19	2.615	25.47	2.444	26.95	7.25	3.73	9.73	5.00
Quartzites:									
Medina	6	2.629	1.44	2.516	10.53	14.84	4.18	15.05	4.24
Baraboo	22	2.650	.13	2.490	3.62	10.26	6.39	10.28	6.40
Alabama	26	2.642	.00	2.510	4.55	9.95	4.93	9.95	4.93
Homewood sandstone	14	2.620	6.49	2.493	14.52	14.50	4.38	15.51	4.68
Montana	28	2.641	.92	2.475	6.88	12.18	6.30	13.32	6.37
Soft Medina	37	2.615	2.25	2.573	9.32	9.26	1.34	9.47	1.36

TABLE 8—Continued.

Materials	No.	1500° C							
		Before		After					
						In terms of original exterior volume		In terms of original solid volume	
		Specific gravity	Per cent porosity	Specific gravity	Per cent porosity	Per cent exterior volume expansion	Per cent solid volume expansion	Per cent exterior volume expansion	Per cent solid volume expansion
Raw brick mixes:									
Medina	6	2.603	26.48	2.323	33.57	22.23	7.65	30.23	10.40
	4	2.640	26.03	2.333	35.85	28.50	8.28	38.50	11.20
	8	2.626	30.00	2.328	39.30	27.52	7.36	39.30	10.51
Homewood sandstone	14	2.615	28.20	2.336	35.70	22.90	6.83	31.70	9.47
Baraboo	19	2.606	24.79	2.344	31.38	19.65	6.92	26.11	9.20
Quartzites:									
Medina	6	2.635	5.60	2.303	26.76	46.70	12.00	49.40	12.70
Baraboo	22	2.650	.27	2.293	10.95	29.30	15.41	29.43	15.47
Alabama	25	2.640	.02	2.295	11.12	29.20	14.86	29.20	14.86
Homewood sandstone	14	2.630	6.52	2.326	26.64	43.40	11.66	46.40	12.50
Montana	28	2.638	1.05	2.306	22.39	45.30	13.83	45.80	13.87
Soft Medina	37	[a] 2.602	[a] 2.01	[a] 2.403	[a] 14.84	[a] 24.16	[a] 7.56	[a] 24.62	[a] 7.70

[a] In separate burn, not comparable to other figures.

In Table 8 are shown the specific gravities and porosities of raw brick mixes and quartzites before heating, their specific gravities and porosities after heating; also exterior and solid volume expansions after heating in terms of both original exterior volumes and original solid volumes.

The exterior and solid volume expansions in terms of the original exterior volumes show the behavior of any one material, and these same expansions in terms of the original solid volumes enable us to compare different materials directly. The specific gravities of the burned pieces show that up to 1450° C the brick mixes were transformed much more than were the quartzites, but that with the 1500° C heating the quartzites reached lower specific gravities than did the brick mixes. This may be due to formation of high specific gravity glass in the brick mixes. The specific gravities of the quartzites after burning show that all varieties were transformed at practically the same rate and that the 1500° C heating was sufficient to produce approximately minimum attainable specific gravity. These facts hold for the Homewood sandstone as well as for the more highly metamorphosed quartzites.

Considering the volume expansions in terms of the original solid volumes, we see that the solid volume expansions of the quartzites for the 1500° C burn is greater than that of the brick mixes. This agrees with the fact that the specific gravities of these quartzites which were burned to 1500° C is lower than that of the corresponding brick mixes when similarly burned. The exterior volume expansions of the Homewood sandstone and Medina and Montana quartzites are much greater than those of Baraboo and Alabama quartzites. This larger expansion in the case of Homewood and Medina is probably due to their structure being slightly looser than that of Alabama and Baraboo. In the case of Montana quartzite this difference may be due to its being much finer grained than any other materials tested. These phenomena apparently also hold for the brick mixes; thus, brick made from Baraboo or Alabama quartzites would not be expected to increase as much in porosity during burning as would those made from the other varieties shown in this test. This agrees with our other data on this subject.

(*e*) *Series 5.*—In connection with added ingredients the results of the first test are given in Table 9. These figures show that a much greater transformation took place in the case where lime plus shale was added than where shale alone was added. It is also apparent that 2 per cent of shale in conjunction with lime produced considerably more transformation than did 1 per cent of Fe_2O_3 in conjunction with lime.

TABLE 9.—Effects of Added Ingredients, Judged by the Percentage Exterior Volume Expansion of Various Mixtures, Which Were Heated Twice to 1400° C in Terms of Original Exterior Volume

Material	No.	Per cent volume expansion, first burn to 1400° C	Per cent total volume expansion after second burn to 1400° C	Per cent expansion caused by second burn
Medina Quartzite plus 2 per cent CaO plus 1 per cent Fe_2O_3	1	10.82	11.82	1.00
Medina Quartzite plus 3 per cent Metropolitan shale	2	4.48	5.80	1.32
Medina Quartzite plus 2 per cent CaO plus 2 per cent Metropolitan shale	3	11.16	13.45	2.29

In Table 10 are shown the results of the second test. In general it may be said that the ingredients added in this test, other than lime, did not appear to alter the rate of transformation or porosity change greatly at the temperature used (1450° C). Lime did, however, greatly increase the rate of transformation and larger percentages apparently increased the rate still more. This speed-

ing up of the rate of transformation apparently causes greater increase in porosity than does a corresponding amount of transformation at a lower rate.

TABLE 10.—Effects of Added Ingredients, Judged by Specific Gravity, Porosity, and Exterior Volume Changes of Various Mixtures Which Were Heated to 1450° C

Materials	No.	Before heating		After heating		Per cent exterior volume expansion
		Specific gravity	Per cent porosity	Specific gravity	Per cent porosity	
Medina plus 2 per cent CaO	1	2.615	38.4	2.405	40.20	9.60
Medina	2	2.615	2.2	2.573	9.32	9.25
Medina plus 3 per cent Metropolitan shale	3	2.530	37.0	2.520	43.50	6.30
Medina plus 2 per cent Tennessee ball clay	4	2.610	40.4	2.568	42.70	4.32
Medina plus 2 per cent CaO plus 2 per cent Metropolitan shale	5	2.630	36.7	2.389	38.80	11.80
Medina plus 2 per cent CaO plus 2 per cent Tennessee ball clay	6	2.630	38.8	2.427	41.00	9.77
Medina plus 2 per cent CaO plus 1 per cent Al_2O_3	7	2.630	39.1	2.468	41.70	9.10
Medina plus 2 per cent CaO plus 2 per cent Al_2O_3	8	2.640	37.6	2.496	39.20	5.94
Medina plus 0.5 per cent CaO plus 1.5 per cent CaF_2	9	2.560	35.3	2.413	40.20	9.49
Medina plus 2.5 per cent CaO	10	2.615	30.9	2.389	33.70	11.53
Glass sand plus 2 per cent CaO	11	2.620	33.2	2.562	36.02	4.53

Table 11 shows the results of the third test (four-day burn). The coarse grind which the material received does not appear to have materially altered the properties of the burned pieces. The effects of pressure were readily discernible, however, as characteristic numerous fine cracks over the surface of all the pieces after burning. In this test all the pieces approximately reached minimum specific gravity during burning. However, a decided effect of the sodium chloride is shown by the exceptionally great increase in porosity on burning of the mix containing it.

TABLE 11.—Effects of Added Ingredients and Molding of Brick Under Pressure, Judged by Specific Gravity, Porosity, and Exterior Volume Changes of Various Mixtures, Which Were Heated to 1450° C in Four Days

Materials	No.	Before heating		After heating		
		Specific gravity	Per cent porosity	Specific gravity	Per cent porosity	Per cent exterior volume expansion
Medina plus 2 per cent CaO aepressed 5000 pounds per square inch	1	2.630	28.10	2.321	35.80	24.10
Medina plus 2 per cent CaO plus 2 per cent Metropolitan shale 5000 pounds per square inch	2	2.630	27.52	2.321	34.90	24.23
Medina plus 2 per cent CaO plus 1.4 per cent NaCl 5000 pounds per square inch	3	2.640	27.74	2.312	41.30	.....

In considering all the tests that have to do with added ingredients, the indications are that rate of transformation is increased by lime or sodium chloride, very slightly increased by impure ferruginous clay when used in conjunction with lime, and is practically not affected by pure plastic clay or the other ingredients which were tested. In this connection it must be remembered that the above tests are special cases and that in nature the foreign ingredients would in all probability be much more intimately associated with the quartz grains and thus cause somewhat different results.

In connection with the tests of calcined material a microscopic examination of the quartzites after the second calcination revealed the fact that they were largely converted to cristobalite, but that small interior portions still remained as unaltered quartz. In the first of these tests the Medina and loose-grained Medina mixes crumbled and fell apart during burning, while the Baraboo mix held together, but had no appreciable strength. In the second test, in which the test pieces were molded under a pressure of 5000 pounds per square inch, the results were the same. One piece of Baraboo mix was found to be strong enough after burning to allow of its volume being measured. This showed an exterior volume increase of approximately 3 per cent.

The above data indicate that strong brick can not be made from strongly calcined material, at least when the duration of burning of the brick is only a few days. However, the permanent exterior volume increase of such material is small.

(*f*) *Series 6.*—The materials of the first burn of the test of soft Medina quartzite contained 2 per cent of lime. It was found that 91.6 per cent of the 8-mesh quartzite went through a 20-mesh screen before being mixed with the lime. Apparently the 8-mesh material yielded a considerably more durable brick than the 10-mesh grind. However, all brick of this burn were too friable to be termed first class.

The results of the later tests of this series are shown in Table 12. As is shown under burn No. 3, the 1 per cent of lime plus 1½ per cent of Tennessee ball clay bricks expanded more than did the ones where lime alone was used, and the bricks which contained 2.5 per cent of Tennessee ball clay alone expanded less than the bricks that contained lime.

TABLE 12.—Effects of Added Ingredients, Judged by Linear Expansion of Various Mixtures, Which Were Given Heat Treatments of Varying Duration and Temperature

No.	Materials	Burn No.	Piece No.	Through mesh	Cone No.	Maximum temperature	Per cent linear expansion	Total per cent linear expansion
37	Medina previously calcined to 1300° C 2 per cent CaO.	2	[illegible]	8	17	1485 flash	3.15	.....
37	Medina plus 2.5 per cent CaO	3	3	[illegible]	16+	1485 flash	3.77	.....
37	Medina plus 3.0 per cent CaO		4	[illegible]			3.72	.....
37	Medina plus 1.0 per cent CaO plus 1.5 per cent Tennessee ball clay.		5	[illegible]			4.07	.....
37	Medina plus 2.5 per cent Tennessee ball clay.		6	8			3.69	.....
		4	2	.....	.....	1450° C for 5 hours	1.70	4.85
			3				1.85	5.62
			4				2.11	5.83
			6				2.28	5.97
37	Medina plus 2.5 per cent CaO	5	7	8	20	1470° C for 3 hours	5.40	.....
37	Medina plus 2.5 per cent CaO		8	20			6.33	.....
39	Hard quartzite plus 2.5 per cent CaO.		9	8			7.32	.....

In the fourth burn mixes Nos. 3, 4, and 6 (Table 12) approached a common percentage expansion, while the expansion of mix No. 2 was considerably less. This seems to indicate that in this burn the materials reached approximately the limit of expansion, and that this limit was decreased in the case of No. 2 by the amount of transformation that had taken place in the calcining of the quartzite. Burn No. 5 shows a decided difference in expansion between the 8-mesh and 20-mesh material. The 20-mesh material after burning had no appreciable strength. This lot of brick, however, were made up somewhat drier than the 8-mesh brick. This may have had something to do with their friableness.

The hard rock mix No. 9 shows a greater expansion than the soft rock tested. It yielded a brick which compared favorably with commercial brick as far as ring and spalling tendency are concerned.

In the several tests above with loose-grained Medina quartzite, the exterior volume expansion did not exceed 6 per cent except with the most friable brick. Although several combinations gave fairly satisfactory brick, the most satisfactory probably being 8-mesh quartzite plus 2.0 per cent of lime. None of them had the best ring and all showed a decided tendency to spall.

(*g*) *Series 7.*—Table 13 gives the data of the burn in a commercial kiln. Nos. 1, 2, and 3 are the same mixes as Nos. 1, 2, and 3 of Table 11, and received the same pressure in molding. No. 4 was a mix from some of the commercial Medina which had been calcined twice to 1500° C. This piece was subjected to 5000 pounds per square inch when molded. No. 5 was a mix made from the clacined Baraboo quartzite.

TABLE 13.—Effects of Added Ingredients, Molding of Brick Under Pressure, and the use of Calcined Quartzites, Judged by Specific Gravity, Porosity, and Exterior Volume Changes of Various Mixtures, Which Were Heated to Cone 16+ in 14 Days in a Commercial Kiln

Materials	No.	Before heating		After heating		
		Specific gravity	Per cent porosity	Specific gravity	Per cent porosity	Per cent exterior volume expansion
Medina plus 2 per cent CaO, repressed 5000 pounds per square inch	1	2.642	29.27	2.362	31.05	14.83
Medina plus 2 per cent CaO plus 2 per cent Metropolitan shale, 5000 pounds per square inch	2	2.640	27.82	2.347	31.10	16.20
Medina plus 2 per cent CaO plus 1.4 per cent NaCl, 5000 pounds per square inch	3	2.640	28.03	2.313	39.02	36.20
Medina strongly calcined plus 2 per cent CaO	4	2.310	31.64	2.310	35.00	2.48
Baraboo strongly calcined plus 2 per cent CaO	5	2.310	29.25	2.283	30.30	1.28

The specific gravities of 1, 2, and 3 after burning show that the material containing the shale and lime were transformed somewhat more than that containing lime alone, and that the material which contained sodium chloride and lime was transformed considerably more than either of the other two. Its porosity increased to practically double that of either of the others. Its structure was so friable that it barely held together while being measured. It could be crumbled readily between the fingers, whereas the other two were moderately strong.

Comparing the porosities of 1, 2, and 3 with 1, 2, and 3 of Table 11 we see that apparently the former had appreciably lower porosities than the latter, also inspection showed that pieces Nos. 1 and 2 of the latter were much more friable than those burned in the commercial kiln. The fine surface cracks, apparent in the repressed material of the short-time burn, were also present in the commercially burned pieces. The pieces present in this commercial burn, which were made from highly calcined quartzites, showed very little volume increase, and had practically no strength when

burned, which facts bear out what was found to be true for a short-time burn.

(*h*) *Series S.*—Table 14 shows the changes in specific gravity and porosity brought about in the underburned, medium burned, and well-burned bricks, made from Medina quartzite by rapid reheating.

TABLE 14.—Effects of Rapid Reheating to 1480° C, on Underburned, Medium Burned, and Well-Burned Brick, Made of Medina Quartzite

Materials from one kiln[a]	No.	Before heating		After heating to 1480° C		
		Specific gravity	Per cent porosity	Specific gravity	Per cent porosity	Condition of brick
Medina brick No. 6:						
Soft burn	1	2.50	30.1	2.32	39.6	Cracked.
Medium burn	2	2.46	27.9	2.32	36.4	Do.
Hard burn	3	2.32	33.5	2.33	34.1	Fairly sound.

[a] Effects judged by changes in specific gravity, porosity, and amount of cracking.

In the first place this table indicates what a wide range in the degree of burning may occur in a single kiln of bricks. It also indicates that in a moderately burned brick the porosity is lower than in either underburned or well-burned brick. The high porosity and great decrease in specific gravity of brick No. 1 after heating shows what rapid heating does to underburned brick; similarly, No. 2 shows the undesirable effect produced in a medium burned brick; and No. 3 shows what a relatively small porosity change is occasioned in the case of a well-burned brick. This evidence is strongly in favor of a brick in which the silica has been largely transformed from quartz to the other crystal forms.

2. PROPERTIES OF FINISHED BRICK AND DATA ON MANUFACTURE

(*a*) *Data on Commercial Bricks.*—When it was once determined that the specific gravity of a silica brick was a definite measure of the degree to which the brick had been burned, it became desirable to know what the degree of accuracy of such measurement is, whether all varieties of material would fall within these limits, and what the average specific gravity of the leading varieties of brick is.

The specific gravity of raw quartzite is approximately 2.650 and the specific gravities of artificial cristobalite and tridymite are approximately 2.333 and 2.270, respectively. Hence the

specific gravities of brick should lie between these two extreme points. The figures of Table 15 represent the specific gravities of 12 pieces of raw American quartzites.

TABLE 15.—Check Specific Gravity, Determinations made on Quartzites by the Wet, Dry, and Suspended Weight Method, to Determine the Degree of Agreement Between Separate Determinations by this Method, Using a Vacuum Equal to 24 Inches of Mercury, to Insure Saturation

	Group 1	Group 2
Close-grained material	2.650	2.650
	2.650	2.650
Mean	2.650	2.650
Open porous material	2.620	2.604
	2.610	2.600
Mean	2.615	2.602
Close-grained material	2.646	2.642
	2.638	2.638
Mean	2.642	2.640

Two of these varieties are nearly nonporous and the third is an open-porous variety. Each set of two determinations for a variety were made on the same day, but the different groups for a variety were determined at different times. The degree of agreement of the determinations for any one variety can be seen from the figures. Considerably over a thousand such determinations have been made with quartzites, bricks, etc., and the degree of agreement of the determinations has been quite generally as close as the ones presented. A series of specific gravity determinations were also made on these quartzites by means of a picnometer, and were found to check the above results as closely as the individual picnometer readings for a variety checked each other.

To determine whether all varieties of material would be likely to fall within these limits a number of unusual varieties of material were tested. The results of these tests are shown in Table 16.

TABLE 16.—Properties of Some Unusual Silica Brick Materials

Materials	No.	Specific gravity	Per cent porosity
Medina quartzite brick in kiln with Baraboo draw trial burn 1 day at 1500° C, 4 days at 1450° C+	6	2.321	29.45
Medina quartzite brick heated 40 times on bag wall of test kiln to 1450° C	6	2.275	31.85
Brick made from Baraboo quartzite which received 9 years' service in Koppers by-product coke oven	18	2.314	19.94
German brick, Stella Werke* * *	33	2.372	20.07
Homewood sandstone	14	2.626	6.52
Brick made from Homewood sandstone	15	2.448	28.95
California quartzite plus California River sand plus 2 per cent of CaO; 2 heatings in commercial clay brick kiln	34	2.437	30.02
Raw Indiana chert rock	29	2.585	1.83
Brick made from Indiana chert	29	2.273	25.64
Alabama mica quartz schist	35	2.690	4.52
Brick made from mica quartz schist	35	2.616	34.07
Brick made from slightly impure silica sand plus 8 per cent CaO $(OH)_2$	36	2.527	30.40
Brick made from Medina quartzite that received 10 commercial burns to cone 14	6	2.271	27.35
Brick made from West Virginia quartzite	38	2,270	28.40

From this table we see that the specific gravities of products which have seen service at high temperatures may be considerably above the theoretical limit, and even appreciably above the specific gravities of some unused bricks made from Medina quartzite. Likewise, the German-made brick, the Homewood sandstone and the brick made from it, as well as the brick made from a mixture of quartz and California river sand are well within the limits. The Indiana raw chert rock is of interest because its specific gravity is considerably lower than that of quartz. Likewise, the specific gravity of the resultant brick is near the lower limit. These matters concerning chert, however, are entirely in accordance with the work of Mellor, given above, which shows that the original specific gravity of this kind of material is approximately 2.61 before heating, and reaches 2.22 as a final limit when repeatedly heated to cone 10, and also that the rate of transformation of this material is much more rapid than that of quartz, when heated to the same temperature. This view with respect to the rate of transformation of chert is also borne out by the work of Khoctsky and Rickie and Endell, also cited above. The mica quartz schist and the brick made from it are of interest in that they both have a specific gravity above 2.65 on the opposite side of the permissible range from chert. This is to be expected, however, from the fact that mica (probably muscovite in this case) has a specific gravity somewhere between 2.70 and 3.10.

This particular material is outside the range of usefulness for silica brick, however, as its softening temperature is too low.

Some question has been raised as to the necessity of a vacuum for four hours in saturating pieces for specific gravity and porosity determinations. Hence the tests shown in Table 17 have been carried out by obtaining saturation by boiling in water for four hours without subjecting to a vacuum.

TABLE 17.—Check Specific Gravity Determinations Made on Burned Silica Bricks, by the Wet, Dry, and Suspended Weight Method to Determine the Degree of Agreement Between Separate Determinations, when Saturation is Obtained by Boiling in Water for Four Hours

Materials	Approximate size and weight of piece							
	2-inch cube, 260 g, 4 hours in boiling water		1.25-inch cube, 65 g, 3 hours in boiling water		0.75-inch cube, 18 g, 2 hours in boiling water		0.5-inch cube, 10 g, 1 hour in boiling water	
	Specific gravity	Per cent porosity	Specific gravity	Per cent porosity	Specific gravity	Per cent porosity	Specific gravity	Per cent porosity
No. 6. Brick made from Medina quartzite, soft burn	2.47	29.0	2.48	29.2	2.51	28.8	2.49	30.4
	2.49	28.4	2.49	28.3	2.49	29.2	2.48	29.1
No. 6. Brick made from Medina quartzite, well burned, good ring	2.31	28.9	2.33	29.0	2.34	28.7	2.32	28.0
	2.30	28.7	2.33	29.2	2.33	26.3	2.32	27.0
No. 28. Brick made from Colorado quartzite	2.43	26.3	2.43	27.0	2.44	27.7	2.41	26.8
	2.40	26.1	2.43	26.9	2.44	27.5	2.42	26.8
No. 29. Indiana chert brick	2.27	24.8	2.29	24.1	2.30	24.2	2.28	24.0
	2.27	24.1	2.28	23.0	2.30	23.8	2.28	22.5

For the range of materials tested the degree of agreement appears satisfactory for our purpose. As far as tested these results also appear to check those obtained by use of the vacuum. However, wherever practicable it is safest to use the vacuum.

In making specific gravity and porosity tests on a commercial brick it is well to take two pieces for test, from different parts of the brick. If they do not check, it is well to repeat the test to determine whether the difference is due to error or to actual variations in the brick. It is advisable always to have the pieces large enough so that the presence of large grains will not cause errors in porosity.

Considering Table 18, we see that the average specific gravity of all varieties of brick tested is 2.384, and the average porosity is 26.35 per cent. The average specific gravity shown by commercial brick made from Medina quartzite is 2.357, and the lowest porosity shown is 2.291. The porosities of Medina brick range from 23.10 to 31.52. The average specific gravity of brick made from

Baraboo quartzite is 2.399 and the lowest specific gravity of a single-burn brick is 2.381. The porosities of Baraboo brick range from 23.45 to 25.15.

TABLE 18.—Specific Gravities and Porosities of some of the Chief Brands of Commercial Silica Brick Manufactured in the United States, including a list of the Quartzites From Which They Were Made

Quartzite from which brick were made	No.	Specific gravity	Per cent porosity
Medina (Tuscarora)	1	2.375	27.15
	2	2.296	25.84
	3	2.424	28.52
	4	2.393	27.16
	5	2.340	22.80
	6	2.291	26.35
	7	2.375	28.35
	8	2.336	31.52
	9	2.321	25.97
	10	2.358	26.10
	11	2.291	29.56
	12	2.480	23.10
	13	2.340	29.88
Average		2.357	27.07
Homewood sandstone	14	2.496	30.58
	15	2.448	28.95
	16	2.517	26.55
Average		2.470	28.69
Oneida	17	2.274	25.66
Baraboo (No. 20 made from lightly calcined quartzite)	18	2.381	23.91
	19	2.430	25.15
	20	2.325	31.41
	21	2.390	23.45
	22	2.395	24.79
Average		2.399	24.30
Quadrant formation (Montana)	23	2.495	23.22
	24	2.490	22.66
	25	2.537	22.96
Alabama, probably Weisner Formation	26	2.311	29.75
Dakota and Comanchian (Colorado)	27	2.393	24.63
	28	2.387	24.51
Indiana chert (Mitchell formation)	29	2.273	25.64
Saint Louis, Mo., district: Crypto crystalline silica	30	2.363	25.73
Saint Louis, Mo., district: Chalcedony bonded sand stone	31	2.393	24.70
Eastern Pennsylvania (probably Chicke's)	32	2.335	26.85
Grand average		2.384	26.34

With other varieties of material the brands of brick are too few to be of value for general conclusions. It is of interest to note that in the case of the chert brick, the specific gravity is 2.273 and

the porosity 25.64 per cent. The data concerning time and temperature of burning during manufacture of the bricks represented in this table are so incomplete and inaccurate that their presentation would only cause confusion. However, the data at hand indicate that bricks made from qnartzite and having a specific gravity below the average (2.384) have been burned from cone 18 to 20.

The above figures can be considered representative of silica refractories, as they are manufactured in the United States at the present time.

(*b*) *Analyses and Softening Temperatures.*—A typical analysis [24] of brick made from Medina quartzite is as follows:

	Per cent
SiO_2	96.25
Al_2O_3	.89
Fe_2O_3	.79
CaO	1.80
MgO	.14
Alkali	.39
	100.26

Brick having an analysis such as this should have a softening temperature somewhat above that of Orton cone 32.

Brick No. 15, of Table 16, which was made of Homewood sandstone, has a softening temperature above that of cone 32, and brick made from Baraboo quartzite usually have softening temperatures above that of cone 31.

A German silica block (Stella Werke * * *) has an analysis as follows:

	Per cent
SiO_2	94.20
Al_2O_3	1.60
Fe_2O_3	1.62
CaO	2.24
MgO	.09
	99.75

Its softening temperature was found to be somewhat above that of Orton cone 30.

[24] K. Seaver, Manufacture and Tests of Silica Brick for the By-Product Coke Oven, Am. Inst. Min. Eng. Transactions, 53, pp. 125–139; 1916.

Analyses of the raw Indiana chert and of brick made from it are as follows:

	Chert	Brick		Chert	Brick
	Per cent	Per cent		Per cent	Per cent
SiO_2	96. 67	95. 60	K_2O	0. 16	0. 16
Fe_2O_3	. 48	. 61	Na_2O	. 05	. 06
Al_2O_3	1. 59	1. 80	Ignition	1. 28	. 12
CaO	. 07	2. 01			
MgO	Nil	Nil		100. 28	100. 36

The softening temperature of the brick is somewhat above that of cone 30.

The analysis of a brick (No. 36, Table 16) made from slightly impure silica sand and 8 per cent of hydrated lime is as follows:

	Per cent
SiO_2	90. 44
Al_2O_3	. 94
Fe_2O_3	1. 10
CaO	6. 50
MgO	. 81
Na_2O	. 11
K_2O	. 16
Ignition	. 12
	100. 18

The softening temperature of this brick is the same as that of Orton cone 31.

Brick made from the Quadrant Formation (Montana) quartzite had a softening temperature somewhat above that of cone 30, and the Colorado quartzites also yielded brick with approximately this softening temperature. Tests of other lots of brick made from these Montana and Colorado materials have shown softening temperatures somewhat higher than these, but in each case they were below that of cone 32.

With reference to the effect of high iron content, it is interesting to note that material No. 10, Table 18, contains 1.60 per cent of Fe_2O_3 and its softening temperature is above that of cone 32. This iron is present as grains of iron oxide. This is in accordance with the work of those [25] who found 15 to 17 per cent of magnetite in the darkened glassy surface portion of brick that had become "seasoned" through use in a steel furnace. The seasoning process apparently made them less subject to injury from changes in temperature, etc.

[25] Silica and Refractory Material, Cosmo Johns, Trans. of the Faraday Society, 12, parts 1, 2, and 3.

Material No. 18, Table 16, which was made from Baraboo quartzite, received nine years' service as part of the wall of a Koppers by-product coke oven. It was between the coking chamber and a heating flue, 20 feet from the pusher end and 6 inches above the floor of the coking chamber. When the oven was in operation, the average temperature on the coking side of the brick was 1110° C (2000° F), the average on the heating flue side was 1330° C (2400° F) and the maximum on the heating flue side was 1550° C (2800° F). A sample of the brick that had been in contact with the coke possessed a softening temperature above that of cone 32, while samples from the interior of the brick and the side which was exposed to the heating flue gasses had a softening temperature somewhat above that of cone 31. (See other properties of this material below under (*d*).)

The above analyses and softening temperatures show in a limited way what a variety of substances come within the range of chemical composition of materials suitable for silicia refractories.

It is apparent from these analyses and softening temperatures that appreciable quantities of iron and alumina (up to 1.60 per cent of each) may not greatly lower the softening temperature of a brick. From these analyses it is also apparent that in all cases the total alkalies present must be fairly low (not usually exceeding 0.40 per cent). In all tests where much larger quantities have been present the softening temperature has been found to decrease rapidly with increase of alkali.

(*c*) *Load tests.*—In running load tests on silica brick, it was soon learned that if heated to 700° C at the same rate as is usually followed with clay brick (270° C in 20 minutes, 520° C in 40 minutes, and 690° C in 60 minutes), the specimen invariably spalled. Hence, a rate of 50° C in 15 minutes to 700° C was tried. No spalling occurred. This rate of heating has since been used with many varieties of silica brick and in no case has spalling of the brick occurred. Increasing the rate to 100° C in 15 minutes above 500° C seldom if ever caused spalling.

These tests frequently repeated have lead to the conclusion that practically all spalling of silica brick occurs between atmospheric temperature and 500° C.

A brick of the same variety as No. 27, Table 18, was subjected to a load test of 25 pounds per square inch and a maximum temperature of 1400° C maintained for one and one-half hours, and a record was kept of its change in length by means of two catheto-

meters, one sighted on the base of the brick and one on its top. The results are tabulated in Table 19.

TABLE 19.—Expansion of Commercial Silica Brick No. 27, While Being Load Tested to 1400° C, Under a Pressure of 25 Pounds Per Square Inch

Time in hours	Temperature in degrees centigrade	Per cent linear expansion
0.00	19	0.0
1.00	200	.0
2.50	500	.340
3.00	650	.363
4.50	1200	.408
4.75	1250	.408
5.00	1300	.431
5.25	1330	.431
5.50	1360	.657
6.00	1400	.657
6.50	1400	.657
7.00	1400	.703
7.50	1400	.840
Measured cold after test		.158
Total expansion		.840
Permanent expansion		.158
Difference compared to 0.657 at 1360° C		.682

These figures show that from atmospheric temperature to 300° C a decided expansion took place, and that this maximum point in the curve did not flatten out again until a temperature of approximately 500° C was reached. In other words, approximately one and one-half hours were consumed before this expansion was complete. Above this point there was a small gradual change in length until the maximum temperature (1400° C) was reached, at which temperature a rather decided gradual expansion in length took place. It is also seen that the amount of this latter expansion is practically equal to the amount of permanent expansion of the brick. The figures in this table indicate how difficult and inaccurate it would be to endeavor to express the volume changes of a silica brick caused by heating, in terms of a single thermal expansion factor.

Another of these same brick was subjected to a load test of 25 pounds per square inch and 1500° C as the maximum temperature, which was held for one and one-half hours. In this case the permanent expansion in length was 2.6 per cent, and the load carrying lever arm of the test furnace showed that a decided increase

took place above 1400° C. Other tests of this nature on several varieties of brick have yielded results which are in accord with the above data.

The low temperature volume changes are apparently those caused by the low temperature tridymite inversions (below 200° C), which are accompanied by slight volume changes, and by the Alpha-Beta cristobalite inversion at from 220 to 275° C, which is accompanied by a considerable volume change. The expansion that took place just above 300° C is evidently due to the lag in the penetration of the heat to all parts of the specimen and to the time required for the inversions to take place. Since the percentage of tridymite in most commercial silica brick is quite small, the effect due to the tridymite inversions is apparently negligible. It is also apparent that the slow expansion at high temperatures is due to actual transformation of quartz. In the case of bricks which have been greatly underburned in manufacture, the volume change accompanying the Alpha-Beta quartz inversion at 575° C is apt to cause a slight increase in length at this temperature. These conclusions are in accordance with the results of Fulweiler's work presented above.

(*d*) *Micro Studies.*[26]—Several pieces of the Baraboo draw trial burn (series 3) were examined under the microscope; these included both the quartzite alone and the brick mix. It was apparent from these that cristobalite formed first on the surface of the grains and along cracks through them. The glass present in these pieces had an index of refraction considerably higher than the silica minerals.

This indicates that in the individual grains the transformation of quartz to cristobalite proceeds from the surface to the center, and since high index of refraction indicates high specific gravity in the case of such glasses, it is probable that the presence of much such high index glass may at times tend to cause slightly higher specific gravity than would otherwise be the case.

Material No. 6 (second position) of Table 16, a view of which is shown in Fig. 22, was heated approximately 40 times to an average temperature of 1450° C for short periods. The ground mass is practically all tridymite. The larger particles are either grayish in color or have white centers in a gray outer portion. The white central portions were found to be fine grained and to consist of cristobalite, while the gray material was found to be coarser

[26] A good deal of this information was privately communicated by Dr. C. N. Fenner of the Geophysical Laboratory, Washington, D. C. For other data the writer is indebted to Mr. H. Insley.

grained and to consist of tridymite and a glass of high index of refraction. As shown in Fig. 22, the white spots surrounded by darker bands are the larger particles which show the cristobalite centers surrounded by tridymite and glass.

Consideration of the above studies indicates that the quartz is converted to cristobalite from the surface to the center of the individual grains; that in brick which are mostly cristobalite the ground mass is the first to change from cristobalite to tridymite, and that the larger particles pass progressively from cristobalite to tridymite from the outside to the interior. The latter test shows that even after such an extended heat treatment some cristobalite still remains.

A material from Colorado which is used in small percentages in manufacturing silica brick was found to be a quartz conglomerate bonded with chalcedony, and a material from Missouri (No. 31), which is used alone in the manufacture of silica brick, was found to consist of a medium grained silica sandstone, bonded with a mixture of chalcedony and fine quartz crystals, which crystals are not of the same orientation as the original sand grains.

The Indiana chert was examined and like other cherts it was found to be cryptocrystalline. Scattered through it at wide intervals small groups of quartz crystals were found, which are plainly visible to the naked eye.

The brick made from this chert, and which received the usual burn in a commercial kiln, was found to consist of quartz plus silicate 5 per cent, cristobalite 12 per cent, and tridymite 83 per cent. This brick is of special interest on account of the very large percentage of tridymite which is present in it as a result of a single commercial burn. A comparison of this with the microanalyses of brick made from quartzites shows this plainly.

Material 30, Table 7, is composed of cryptocrystalline silica from southern Illinois and is of interest in that, as far as studied, its behavior is similar to that of chert (No. 29) discussed above, except that much of the southern Illinois rock is apparently composed of grains, practically all of which will pass through a 330-mesh screen, and which are but slightly banded together, while the Indiana chert is much harder and approaches a massive structure.

The German brick (Stella Werke * * *), material No. 33 of Table 16, was examined and found to contain quartz + silicates 20 per cent, cristobalite 78 per cent, and tridymite 2 per cent. This microanalysis in conjunction with the low porosity and high

FIG. 23.—*Commercial brick, Medina quartzite, taken with crossed nicols (magnification 140 diameters), and showing large white patches are quartz grains that have been converted to cristobalite along cracks in the grains; white wedge-shaped particles in groundmass are tridymite twins*

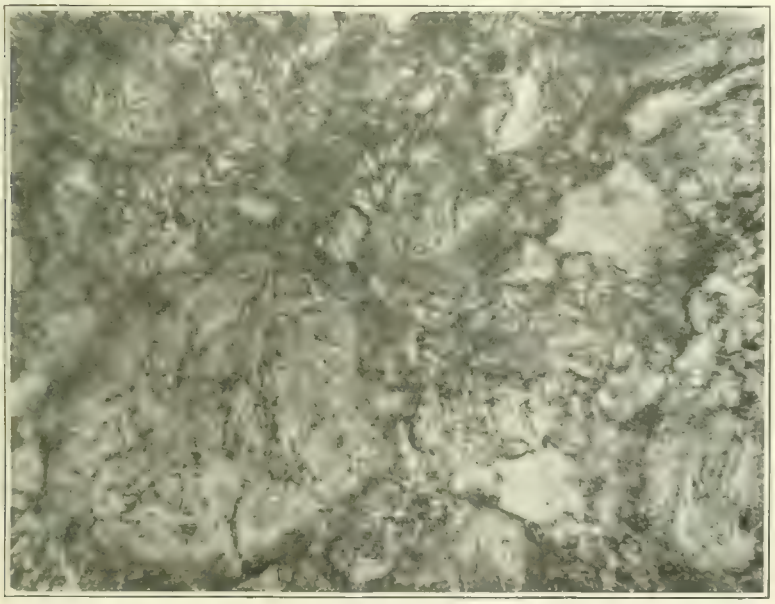


FIG. 24.—*Commercial brick, Medina quartzite, ordinary light (magnification 140 diameters), showing large quartz grains scattered through groundmass of finer material; quartz grains transformed to cristobalite along cracks*

FIG. 25.—*Commercial brick, Baraboo quartzite, crossed nicols (magnification 125 diameters). Large quartzite grains seamed by cristobalite show up well, [illegible]*

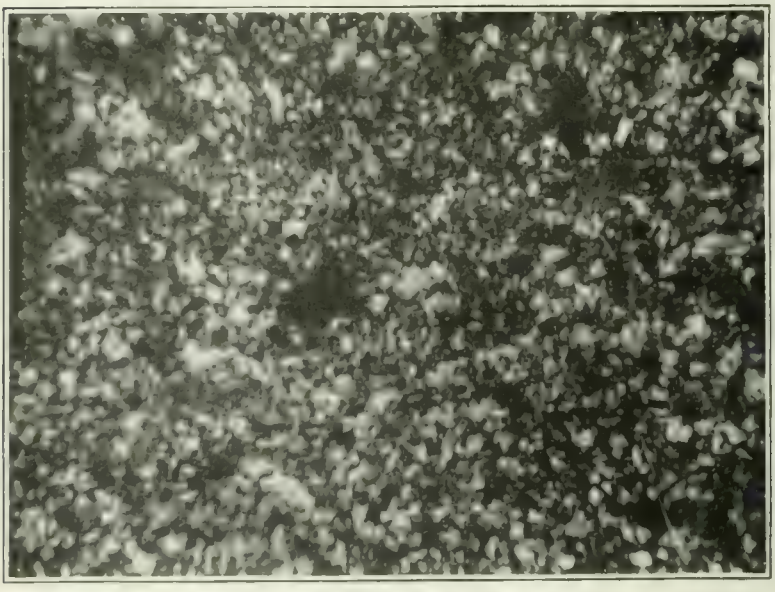


FIG. [illegible] *Iddo's court rock [illegible] (magnification 150 diameters), [illegible]*

FIG. 27.—Commercial brick, Indiana chert, crossed nicols (magnification 140 diameters), mostly tridymite, showing great quantities of tridymite twins

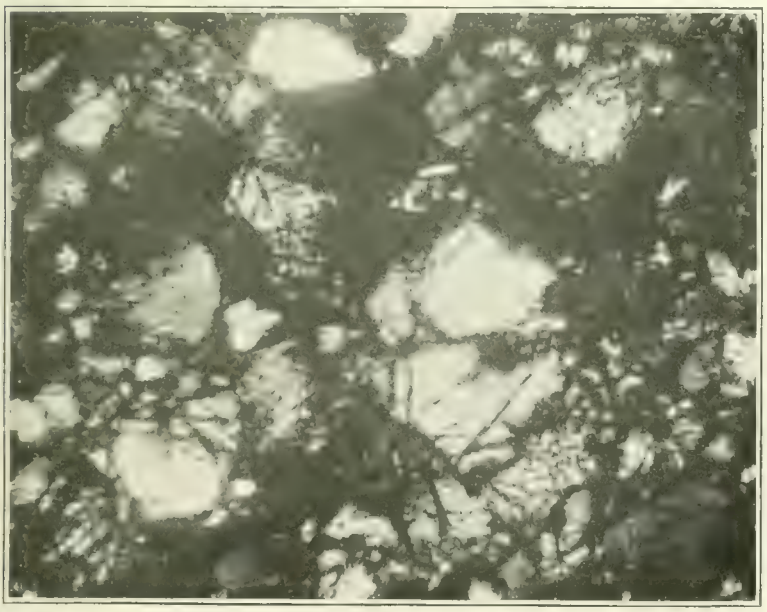


FIG. 28.—*German commercial brick "Stella Werke ☆ ☆ ☆," crossed nicols (magnification 140 diameters), mostly quartz, showing large grains of quartz shot through with cristobalite; practically no tridymite twins*

FIG. 2.—Material of [illegible] heated [illegible] hours. Crossed nicols (magnification 155 diameters). The [illegible] is developed as lace-shaped, tridymite-like [illegible]

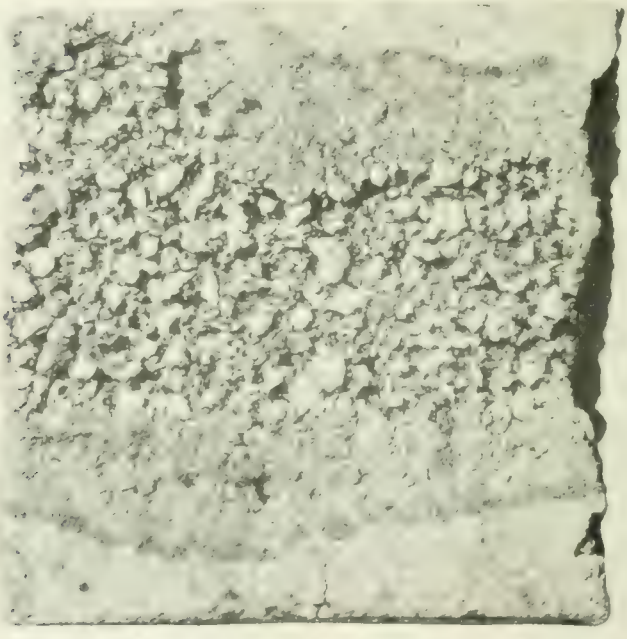


FIG. 3.—Fire-brick of the composition of 370 parts hydrated lime and 100 parts [illegible] heated at 185° C. for one week. Silica [illegible] of fine material, [illegible] of lime to lime

specific gravity of this material indicate that in manufacture it was held for a considerable period at a comparatively low temperature and was not raised to an extremely high temperature during any stage of the burning.

A sample of material No. 18, Table 16, taken from the heating flue side of the brick has a micro composition as follows: Quartz plus silicates 8 per cent, cristobalite 3 per cent, and tridymite 89 per cent. A sample from the central portion of the brick consisted of quartz plus silicates 20 per cent, cristobalite 13 per cent, and tridymite 67 per cent, while a sample from the coke side consisted of quartz plus silicates 31 per cent, crysobalite 24 per cent, and tridymite 45 per cent. This indicates how the percentage of tridymite increases with the severity of the heat treatment from the coking to the flue side and how at the flue side cristobalite is practically absent. In this case the tridymite is somewhat higher and the quartz plus silicates lower than what McDowell has considered as the condition of equilibrium in bricks made from Medina quartzite. (He gave quartz plus silicates equals 13 per cent, and tridymite equals 87 per cent.)

Figs. 23, 24, and 25 are photomicrographs of regular burned bricks made from standard materials, and Figs. 26 to 29 are similar photos of special material and bricks.

In addition to this work, many of the leading quartzites, including the "loose grained" Medina material and other materials of rather unfavorable character were examined with reference to their value for silica brick purposes. To present the results of this study, it seems advisable first to outline the metamorphosis of silica sand to quartzite. The original quartz of most quartzites evidently consisted of rounded grains, with finer siliceous material and impurities, such as hydrated iron oxide in the interstices. During metamorphosis the fragments recrystallize around the grains and with the same orientation as the grains. Silica deposited from solution as quartz may likewise assume the same orientation as the grains. Silica crystallizing in these ways is pure, except for possible occasional inclusions. Hence in well metamorphosed quartzite, impurities such as hydrated iron oxide, which was mixed with the fine grained material in the first place, will be excluded from the grains in recrystallization and will, under the microscope, appear as sharply defined lines between the hard interlocking grains. Where only a small amount of metamorphosis has taken place such impurities will simply be mixed with the fine silica material and the whole will appear as clouded areas of softer

material between the grains of hard quartz. The tightly interlocking grains of a strongly metamorphosed rock tend to yield a conchoidal fracture and show much more mechanical strength than is exhibited by a slightly metamorphosed material.

It has been found that these strongly metamorphosed quartzites are the ones which produce strong bricks, while the materials that show merely cloudy patches between the quartz grains are those which produce very friable brick or are entirely unsatisfactory.

(*e*) *Effect of Slags.*—Fig. 30 shows the effect produced on a silica brick by a slag consisting of hydrated lime 370 parts and bauxite 300 parts, after having been held at 1350° C for one and one-half hours. In this case it is clearly shown that the fine-grained material was fluxed away first, thus leaving an open framework made up of the coarser particles. This shows up plainly by comparison with the part of the brick not touched by slag.

Fig. 31 shows material which has seen service in the crown of a reverberatory copper refining furnace. In this case the slagging material is largely copper oxide. The surface of the brick which has been exposed shows a porous structure somewhat similar to that of Fig. 30, except that in this case the large particles are also quite porous. The other piece shows how such a slag penetrates the large particles of a silica brick, as well as the finer material.

Figs. 33 and 34 show silica brick treated with slags high in copper oxide according to the method outlined by Nesbitt and Bell.[27]

Fig. 32 shows the ends of two bricks which have seen service in the crown of an open-hearth steel furnace. Although this effect is probably due largely to extreme heat as well as slagging, it is of interest, in that it shows how silica brick act in such furnace crowns when the furnace is carried to too high a temperature (stinging the roof). When so overheated the exposed ends of the brick soften and hang down in long stalactitelike strings.

Comparison of the effects of high lime slags and high iron oxide slags when the slagging tests are conducted according to the above-mentioned method, apparently shows that the high lime slag actively attacks the fine material, slagging it away, while the coarser particles remain comparatively unaffected. The slag high in iron oxide, however, tends to penetrate the large grains as well as the finer material, thus altering the brick as a whole. The slagging materials that come in contact with silica brick in furnace work

[27] Loc. cit.

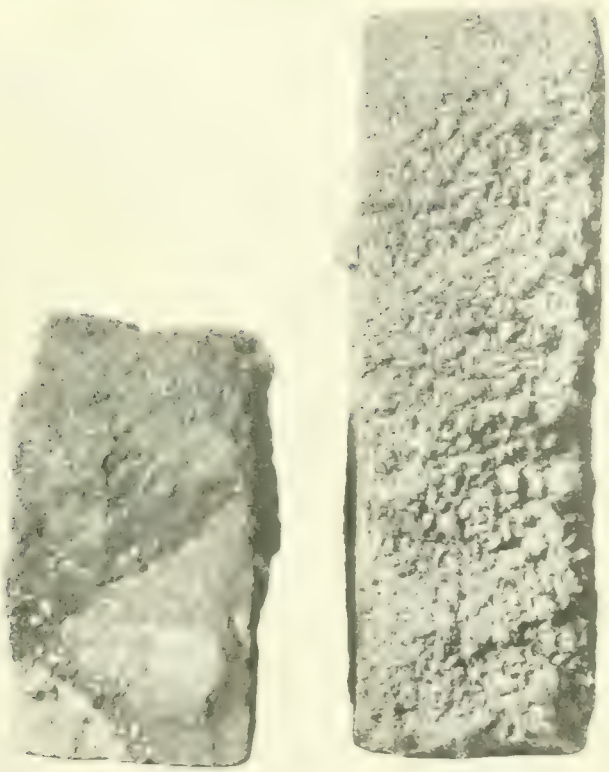
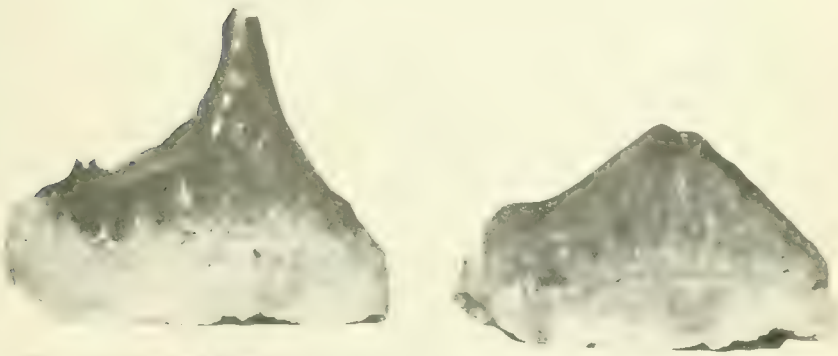


FIG. 31.—*Brick from crown of copper reverberat[illegible] the entire brick. Piece on left shows depth of penetration; piece on right [illegible]*



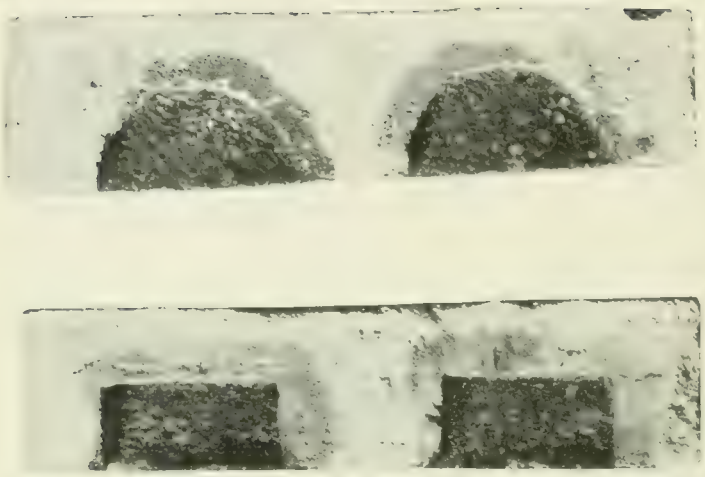


FIG. 33.—*Test made with copper oxide slags by Nesbitte and Bell method, showing nature of surfaces after being acted on by slags. Lower brick was treated with copper reverberatory matting slag, upper brick treated with copper blast furnace slag*

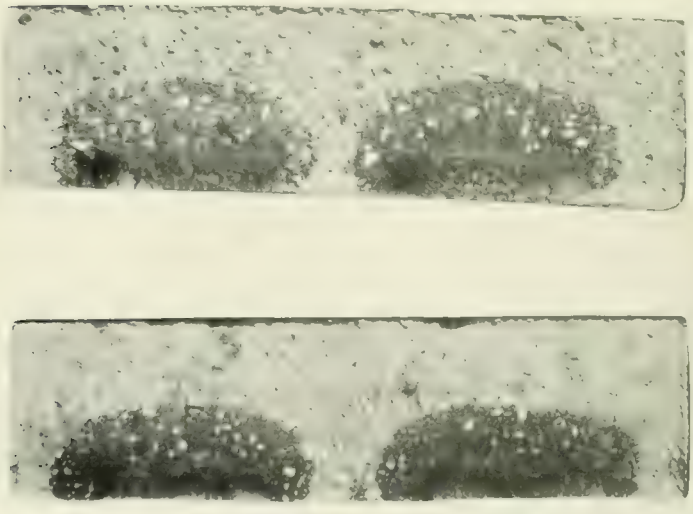


FIG. [illegible] *Same piece and arrangement as shown in Fig. 31, illustrating the amount of penetration of the slags*

are usually of a very basic character, while the brick are extremely acid. Such is the case in steel, copper, and glass furnaces. Such slags will, of course, attack the brick readily. In addition to this many of the slags resulting from such attacking of the brick are very fluid, and have comparatively low melting temperatures. Hence in furnace practice where silica brick come in contact with such basic materials, it is advisable to have the brick as cool as possible by leaving arches, etc., uninsulated.

Slags of this general nature tend first to soak into the fine material of a brick and then gradually into the larger grains. Thus we would expect that the lower the porosity of a brick, the less easily would it be attacked. Also, other things being equal, the greater the proportion of coarse material to fine, the more resistant should be the brick. Likewise quartzites with tightly interlocking grains should be preferable to open-grained material for such purposes.

(*f*) *Screen Analyses.*—Screen analyses and porosities have been determined on 15 unburned silica bricks. These include many of the leading brands and most quartzites that have been used commercially. The data of this work are presented in Table 20.

TABLE 20.—Screen Analyses of Raw Commercial Brick Mixes; Analyses made by the Wet Method

[The porosities of raw and burned bricks of the same brands are also shown]

Screen number mesh	Medina						Baraboo		Homewood formation	
	Number of brick									
	6	2	7	3	5	4	18	21	14	16
	Percentage on screen									
4	1.69	2.44	1.43	1.27	9.97	1.41	3.58	1.72	3.25	11.96
8	19.85	24.18	20.22	14.35	15.63	23.46	27.38	20.98	19.09	
20	11.74	9.67	9.15	10.79	10.01	16.07	15.66	17.86	6.09	6.20
30	3.31	6.36	9.15	9.84	7.76	3.75	5.77	8.84	1.89	4.95
40	10.07	9.67	9.69	15.34	17.23	10.93	4.90	7.97	10.22	29.62
60	18.65	13.97	17.13	16.48	12.17	15.02	12.20	5.73	26.35	15.94
80	1.58	5.70	4.99	3.85	2.97	3.60	2.23	4.24	6.29	2.81
100	3.17	4.26	3.76	3.46	3.27	5.68	3.97	3.22	2.07	5.55
150	4.25	3.33	4.10	2.94	2.32	3.91	3.14	2.60	3.87	1.83
200	3.35	2.45	3.07	2.47	1.51	2.90	2.77	2.76	2.55	1.78
Through 200	9.46	6.63	6.25	5.84	2.88	4.39	8.57	11.27	6.89	8.48
Floated	12.84	11.34	11.06	13.37	14.28	8.90	9.84	12.80	11.38	9.89
Porosity raw brick	26.70	26.15	25.93	27.20	21.22	25.45	23.54	23.20	27.50	28.54
Porosity burned brick	26.35	25.84	28.35	28.52	22.80	27.16	23.91	23.45	30.58	26.55

TABLE 20.—Screen Analyses of Raw Commercial Brick Mixes; Analyses Made by the Wet Method Continued.

Screen number mesh	Colorado		Missouri	Chert	Eastern Pennsylvania	Average of all varieties (per cent)	Theoretical for minimum porosity[a] (per cent)	Diameter of holes between wires, in inches
	Number of brick—Continued							
	28	27	31	29	12			
	Percentage on screen—Continued							
4	0.23	3.27	0.54	0.37	0.39	2.24	0.40	0.18
8	14.17	16.18	11.09	15.47	21.76	18.85	28.10	.093
20	16.55	22.53	18.15	25.00	22.04	14.52	28.20	.034
30	3.25	12.58	9.90	12.14	6.31	7.06	10.30	.0198
40	4.09	10.06	9.80	4.77	6.45	10.72	4.40	.0150
60	12.05	7.09	11.87	7.55	11.57	13.66	6.10	.0087
80	3.62	1.95	3.43	2.37	3.03	3.51	2.50	.0068
100	3.08	4.10	4.09	3.93	3.81	3.83	2.50	.0055
150	3.92	4.40	3.16	2.78	3.32	3.32	2.25	.0041
200	5.04	4.21	3.03	2.48	2.97	2.89	1.75	.0029
Through 200	21.32	4.09	9.57	11.44	9.83	8.46	13.50	.....
Floated	12.65	9.53	15.37	11.66	8.53	11.56		
Porosity raw brick	26.52	22.93	25.30	24.70	20.32	.....	.....	.....
Porosity burned brick	24.51	24.63	24.70	25.64	23.10	.....	.....	.....

[a] Taylor and Thompson, Concrete—Plain and Reinforced, p. 775. Formula for construction of curve is $d=\frac{P^2D}{10\,000}$, where

D=diameter of largest grain.

d=any given diameter.

P=the per cent of mixture smaller than any given diameter. Largest grain=0.182 inch diameter.

The porosities of burned bricks of the same brands are given for comparison with those of the unburned materials.

In general the mixes that have lowest porosities are those that most nearly follow the theoretical curve for minimum interstitial space, as given by Taylor and Thompson. The data show that as a rule raw Baraboo quartzite mixes have slightly lower porosities and more nearly approach this theoretical curve than do Medina quartzite mixes. Material No. 12 appears to most nearly fulfill this condition, and as might be expected this raw mix has the lowest porosity of all materials tested. On the other hand, sandstones and quartzites, which are but slightly metamorphosed, break mostly to individual sand grains, yielding a great excess of these sizes and a brick of high porosity.

In each case there is an excess of material of sizes that correspond to those of the original sand grains that compose the quartzites. The range of grain size lies chiefly between 30 to 60 mesh, inclusive, for Medina, Baraboo, and Homewood quartzites. In

this respect the chert mix more nearly follows the theoretical curve than do most of the quartzites. This is to be expected since, although the chert shows a granular structure, its grains are very minute. In crushing certain of the quartzites, considerable interstitial bonding material adheres to the original sand grains, while with other quartzites a portion of the original rounded grains break free from the interstitial material, and in still other cases the fracture passes directly through the grains. Microexaminations were made of some of the mixes. Thus in the case of No. 6, which material is from Mount Union, Pa., the silica bond clings to some of the rounded grains, but in most cases the fracture has been through the grains. In No. 21, which is from Ablemans, Wis., some grains break free from the bond and show their original rounded form. This is probably due to the thorough filling of the interstitial spaces, resulting in a uniform bond that is not readily torn asunder. In other cases the fracture is through the grains. There are about equal amounts of whole and fractured grains. In No. 14 there is very little material adhering to the grains. What little amount there is appears to be high in iron. The fracture appears to be frequently through the grains. Nearly all the grains are very angular. In No. 28 the fracture is around the grains in most cases rather than through them. A thin layer of cement high in iron clings to the grains. In No. 31, which is the chalcedony bonded quartzite described elsewhere, the fracture appears to be around the grains and not through them. The grains are all quite rounded, a few grains show attached chalcedony, but more of them are nearly completely covered by a cement high in iron. In No. 12, which is probably Medina quartzite, the fracture is usually through the grains. This may account for the low porosity of the mix.

In considering the porosities of the burned bricks we must keep in mind that all bricks have not received exactly the same heat treatment, hence, in this respect the results are not entirely comparable. Comparing the porosities of raw and burned materials it is seen that the porosity of the finished brick depends quite largely upon the porosity of the raw mix.

Inspection of the screened material revealed the fact that for Medina, Baraboo, Homewood, Missouri, and eastern Pennsylvania materials, the original rounded grains, which broke free from the interstitial material, practically all passed through the 30-mesh screen (0.503 mm diam.), and that but very few passed through the 60-mesh screen (0.221 mm diam.), the great

majority being caught on the 40 (0.382 mm diam.) and 60-mesh screens in varying proportions. In Figs. 35 to 40 are shown samples of materials caught on the 40-mesh screen.

At the present time McDowell is carrying on some studies of the sizes of the built-up, interlocking, grains of several quartzites.

(*g*) *Cold Strength Tests.*—In Table 21 the individual cross breaking tests are shown in pounds per square inch.

TABLE 21.—Cross-Breaking Moduli of Burned Commercial Silica Brick; Bricks Set on Edge with 6 In. Between Supports

Test No.	Medina quartzite							
	No. 6	No. 12	No. 11	No. 7	No. 4	No. 1	No. 5	No. 3
1	1557	205	515	616	279	[a] 312	509	372
2	709	242	448	.....	.....	.....	.....	.....
3	452	243	388	.....	.....	.....	.....	.....
4	1003	190	.....	.....	.....	.....	.....	.....
5	863	270	.....	.....	.....	.....	.....	.....
Average modulus	917	230	450	616	279	312	509	372
Effective modulus	[b] 504	197	385	.....	.....	.....	.....	.....
Specific gravity	2.291	2.480	2.291	2.375	2.393	2.375	2.340	2.424

Test No.	Medina quartzite—Contd.		Baraboo quartzite		Homewood sandstone		Oneida quartzite	West Virginia quartzite
	No. 2	[c]	No. 18	No. 21	No. 14	No. 16	No. 17	No. 38 [d]
1	716	.....	565	467	303	329	1049	664
2	.....	.....	593	.....	472	519	.....	1,143
3	.....	.....	544	.....	.....	.....	.....	782
4	.....	.....	546	.....	.....	.....	.....	645
5	.....	.....	527	.....	.....	.....	.....	809
Average modulus	716	[e] 793	555	467	387	424	1049	809
Effective modulus	.....	.....	530	.....	.....	.....	.....	608
Specific gravity	2.296	.....	2.381	2.390	2.496	2.517	2.274	2.270

Test No.	Montana quartzite	Indiana chert	Southern Illinois crypto-crystalline silica	Average all tests
	No. 25 [e]	No. 29	No. 30	
1	423	446	156	.....
2	.....	.....	182	.....
3	.....	.....	.....	.....
4	.....	.....	.....	.....
5	.....	.....	.....	.....
Average modulus	423	446	169	540
Effective modulus	.....	.....	.....	252
Specific gravity	2.537	2.273	2.363	.....

[a] Sheared.
[b] Determined as shown hereinafter.
[c] For "Star Silica Brick" as given by McDowell (loc. cit.).
[d] Porosity, 28.4 per cent.
[e] Machine pressed.

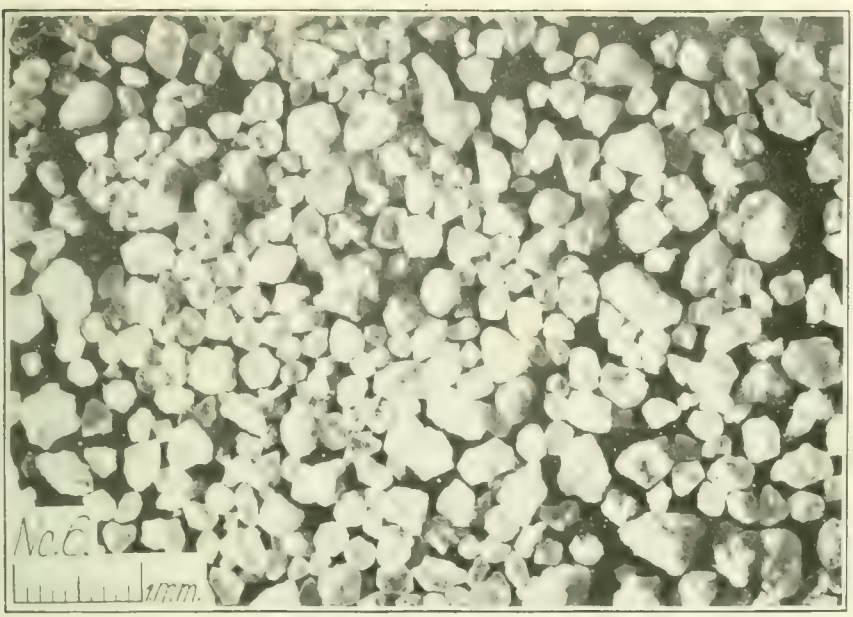


FIG. 35.—*Material on 40-mesh sieve, produced from wet screen analysis of commercial silica brick mix (magnification 25 diameters). Shows considerable percentage of material that has been broken through the interstitial material between the built-up grains. A slight amount of fine-ground material still adheres to the particles*

FIG. 36.—*Screened and photographed same as material of Fig. 35. Shows large percentage of material broken through the grain, also material in which the breaking took place between the original rounded grain and built-up grain. A slight amount of fine-ground material still adheres to the particles*

FIG. 7.—Second enlarged photomicrograph of same material as Fig. 5. Original grains of this material appear more angular than is the case with most of the other quartzites examined

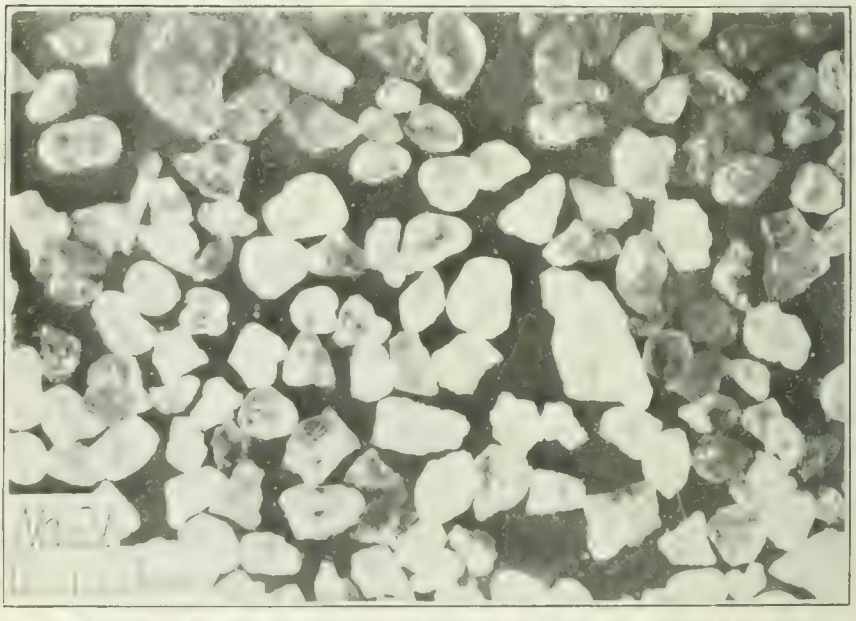


FIG. 8.—Second enlarged photomicrograph of same material as Fig. 5. Shows a large percentage of original rounded grains which have broken free from the material of the built-up grains

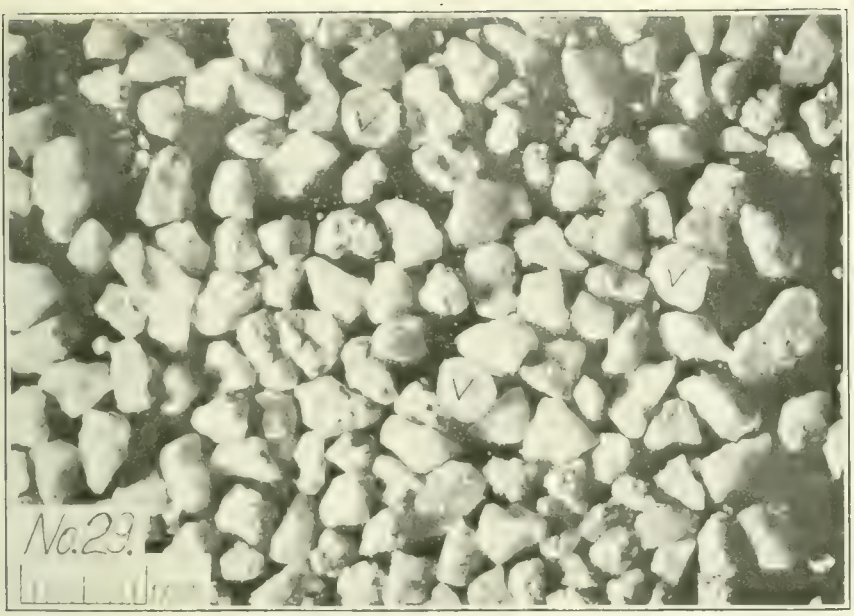


FIG. 39.—Sieved and photographed same as material of FIG. 35. *This chert breaks into angular fragments due to absence of definite grain structure. A slight amount of fine-grained material still adheres to the particles. The 3 grains checked (√) are a different material that was accidentally included.*

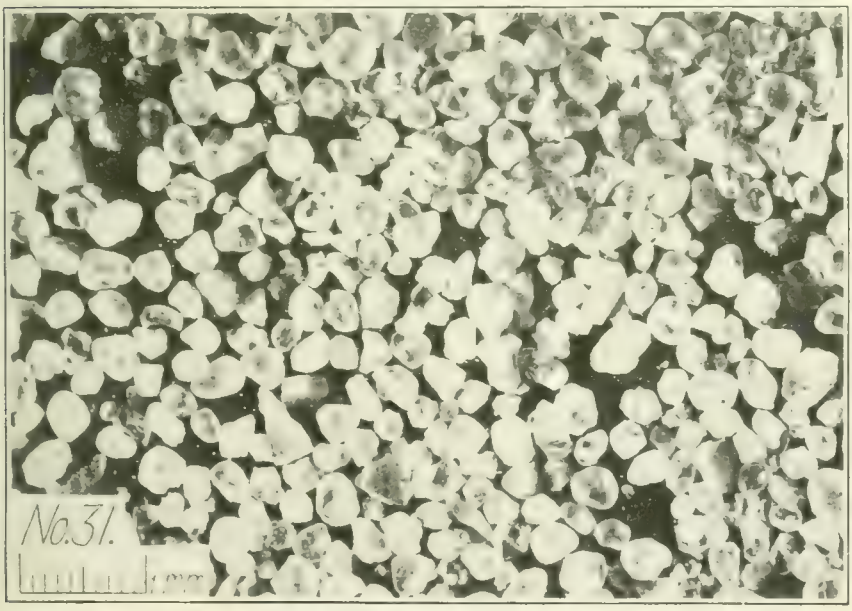


FIG. 40.—Sieved and photographed same as material of FIG. 35. *In this material many of the original rounded grains have broken free from the chalcedony bonding material.*

It is unfortunate but unavoidable that the specific gravity figures given have not in all cases been based entirely on the brick which have been broken, but in several instances include other brick of the respective varieties.

In considering tests of this nature it must be kept in mind that an average of less than five test pieces can not be relied upon as representative of a material.

Comparison of the cross-breaking moduli and specific gravities in Table 21 indicates that usual commercial burned bricks made from quartzite and in which the quartz has been largely transformed to the crystal forms of lower specific gravity, are stronger than those in which there is still a large percentage of unchanged quartz.

It is interesting to compare the great variation in strength shown by the test pieces of variety No. 6 with the comparative uniformity of those of material No. 18. Thus, although the average modulus for No. 6 is much greater than that for No. 18, its effective modulus is slightly less.

A possible explanation of the nonuniformity of materials No. 6 and 38 may be that the difference in degree of transformation may be considerable between the individual specimens.

The unusually low specific gravities of materials No. 17 and 38 almost puts them in a class by themselves; further, both show high moduli of rupture. This table also permits of some comparison between the bricks made from cherty materials (Nos. 29 and 30). If the test on the single specimen of Montana material (No. 25) is any criterion, we might expect to find that this method of machine pressing has a tendency to increase the cross-breaking strength. However, the abnormality of this specimen may be due entirely to the quartzite used.

The brick tested are average samples from many of the leading varieties used for steel furnace work.

73046°—19——5

VI. GENERAL RESULTS

The following statements are based both on the literature cited above and the results of our own work.

1. COMPOSITION[28]

In general silica brick that now give satisfactory service in the United States practically all contain at least 95[29] per cent of SiO_2; many contain at least 1.0 per cent of Al_2O_3, and although few bricks contain much over 1.0 per cent of Fe_2O_3 it has been found that, in certain cases at least, 1.6 per cent did not appreciably depress the softening temperature. Most silica bricks contain 1.60 to 2.0 per cent of CaO. Consideration of the binary system CaO–SiO_2 leads us to believe that up to 6 to 8 per cent of CaO the temperature of softening should not be depressed much below 1600° C. Results of our own work on silica brick containing such percentages of lime lead to the same conclusion.

Apparently the limit of total alkalies in first grade silica brick is approximately 0.4 per cent. Increasing the total alkalies above this pointseems to depress the temperature of softening proportionately. The chemical composition of the brick, however, depends primarily upon the composition of the quartzite from which it is made. The desirability of a quartzite depends not only upon a composition that will yield a sufficiently high softening temperature when made into brick, but also upon its physical character.

2. MICRO STUDIES

(*a*) *Raw Material.*—The order of desirability of the different natural forms of silica, according to European authors, is about as follows:

Amorphous silica, including chert and chalcedony, old quartzites derived from the early crystalline rocks, and vein quartz. Quartz schists, sandstones, and sand are usually too friable or impure to have any value. Most of the silica brick used in the United States are derived from the old quartzites, and hence but little is known

---

[28] So many physico-chemical factors enter into this problem of effect of impurities that at best it is very complex. For instance, intimacy of mixture, rate of reaction between components, melting temperature and fluidity of resulting products, their subsequent dissolving effect on other grains, the time at any given temperature, and the surrounding temperature, all have their bearing.

The data given under this heading are based primarily on analyses of commercial brands that have given the best service, and upon softening temperatures (as described elsewhere herein), on materials of which the chemical composition was known.

The atmosphere in each softening temperature test was apparently neither strongly oxidizing nor strongly reducing. In other conditions prevailed somewhat different results might be expected. Thus the softening temperature test is a laboratory indicator of quality and not an attempt to reproduce what occurs in commercial furnaces.

[29] This statement is based upon the fact that of the 45 varieties of material tested, the unusual materials noted in Table 16 (Nos. 20, 35, and 36) were practically the only ones that contained less than 95 per cent SiO_2.

in this country concerning the other forms. These quartzites vary in desirability in proportion to their degree of metamorphosis. Thus, other conditions being equal, a highly metamorphosed quartzite in which the interstitial material is in sharply defined bands between the hard interlocking quartz grains is apt to be preferable to one in which the untransformed interstitial material is present in cloudy areas between the original rounded grains of quartz sand. Also medium sized or large grains are probably preferable to fine grained material. This is due to the fact that in crushing they do not readily break to as fine a powder as do smaller grains.

The rates of transformation of the different materials to cristobalite on heating in the neighborhood of 1400° C are probably in the order: Amorhpous silica, chert, chalcedony, and quartz glass. These are transformed at about equal speed,which is rapid as compared with that of quartz. Coarse grained quartzites are the slowest to be transformed of any materials used in the manufacture of silica refractories. Fine material and fibrous quartz which have large surface area are transformed appreciably faster than the large quartz grains. This is probably due to the fact that quartz is transformed to cristobalite progressively from the surface inward and along the cracks.

(*b*) *Burned Material.*—On burning silica brick, which contain the usual 2 per cent of lime in commercial kilns, the silica is transformed first to cristobalite and a small amount of glass composed of silica, lime, and the other basic fluxing materials. When approximately 75 per cent of the silica has been converted, tridymite makes its appearance, and on continued burning practically all of the material is eventually converted to tridymite. However, even in the most advanced material where cristobalite has practically disappeared there is still present approximately 10 per cent of quartz and silica glass. In the average commercial burn we may say that as a rule a few per cent of tridymite has been formed and in the case of a Medina quartzite brick there was found to still be small quantities of cristobalite present, even after approximately 40 short burns to an average temperature of 1450° C. It appears probable that Baraboo quartzite mixes are transformed to cristobalite somewhat more slowly than are Medina quartzite mixes, and also that the chert mixes of the material herein considered are transformed appreciably faster than the Medina mixes. The silicate glass formed in silica brick frequently has a considerably higher index of refraction than any of the

other materials in the brick. This high index indicates high specific gravity. Hence it is possible that in certain cases the specific gravity of a well-burned brick may be raised slightly by the silicate glass present.

3. DENSITY

As the specific gravity of quartz is 2.650; that of cristobalite 2.333; and that of tridymite 2.270, it is apparent that on being transformed from quartz to one of the other forms the density of a brick will decrease. It has been found that except for special materials the specific gravity of the burned bricks always falls within these limits—2.650 to 2.270. A material which exceeded the upper limit was a brick made from a micaceous quartz schist. The high specific gravity in this case was undoubtedly due to the mica present, as mica has a specific gravity of approximately 3. However, material containing sufficient mica to thus raise the specific gravity is outside the realm of best quality silica brick, because of low temperature of softening. The chert brick tested were found to have specific gravities in the neighborhood of the lower limit or slightly below it. The lower limit of specific gravity of chert brick is probably 2.230. The location of the specific gravity of a brick between the limits 2.650 to 2.270 definitely determines the degree to which it has been burned. Of the brick tested the average specific gravity of those made from Medina quartzite is 2.357, and the average specific gravity of those from Baraboo quartizite is 2.399, while that of the commercial brick made from chert is 2.273.

The results of series 2 as presented in Fig. 17 show that at the end of the 1500° C burn the specific gravities of Baraboo, Medina and Alabama quartzites were approximately the same and that there are no very great differences in their rates of transformation, but that between 1300 and 1400° C the Medina quartzite appears to be transformed slightly faster than the other two. The average specific gravity (Table 18) of commercial brick made from Baraboo quartzite is somewhat higher than that of brick made from Medina quartzite. Hence since the principal part of the burning received by the average commercial brick is from 1300 to 1400° C it is probable that at these temperatures Baraboo quartzite actually is transformed slightly slower than Medina. As cited above cristobalite is formed progressively from the surface of the grains and along cracks, and hence most rapidly in varieties having the greatest surface area. In accordance with this we should expect the Medina, which is slightly porous, to be transformed somewhat

faster than the more highly metamorphosed Baraboo quartzite, which is practically nonporous. This effect is probably increased by the lime present.

If we concede such a difference in the rates of transformation, Baraboo brick high in unaltered quartz should be less objectionable in by-product coke oven work, where the average temperature is below 1400° C, than underburned Medina brick would be, since the former are less likely to swell rapidly on being heated.

4. VOLUME AND POROSITY CHANGES

(*a*) *Permanent Changes.*—In the burning of commercial silica brick the volume of the solid material actually increases from 10 to 15 per cent of the original solid volume. At temperatures from 1200 to 1350° C brick mixes containing 2 per cent of lime are transformed (drop in specific gravity) much more rapidly than the quartzites alone. Since transformation goes on rapidly at these and higher temperatures, it is practically impossible to duplicate any given degree of burning except near the minimum specific gravity point, in which case the specific gravities of all varieties approach a common value. Hence, to obtain concordant volume-change results from heating tests, it is advisable to use a heat treatment that will insure minimum specific gravity (one to two hours at 1500° C). However, since the average brick contains approximately 27 per cent of pore space, the exterior volume change will be quite different from the solid-volume expansion. It is practically always considerably greater than the expansion of the solid material. Since this is true the porosity of the burned brick is necessarily greater than that of the raw mix.

Strongly bonded quartizites having tightly interlocking grains yield brick that as a rule are low in porosity and show but small increases in porosity upon being burned, while friable quartzites made up of rounded grains yield brick of high porosity which on being burned show considerable increases in porosity. The average porosity of the commercial bricks tested is 26.34; the average of the bricks made from Medina quartzite is 27.07; and the average of bricks made from Baraboo quartzite is 24.30, while that of brick made from chert is 25.64.

The minimum porosity for a given mix will be obtained in burning by raising the temperature at the usual rate to a temperature between 1250 and 1350° C and holding at this temperature for several days. Thus, transformation of quartz will proceed at a moderate rate and the soaking will permit the fluxes present to exert their

maximum effect in drawing the materials together, thus keeping down the increase in pore space to a minimum. To insure thorough transformation, however, the temperature should finally be slowly raised until a heat treatment approximating cones 18 to 20 is obtained. If, however, a raw brick is heated rapidly to a high temperature transformation of quartz goes on rapidly and undue increase in pore space results.

Tests with Medina brick indicate that well-burned brick suffer far less on being rapidly reheated to 1500° C (six hours) than do medium or underburned brick. To compare directly the volume expansions and changes in pore space of different brick mixes and quartzites on burning, however, the solid volumes and exterior volume expansions should be in terms of the original volumes of the solid-volume materials.

(*b*) *Thermal Changes.*—The chief thermal volume changes in commercial silica brick are those due to the tridymite inversions below 200° C and the Alpha-Beta cristobalite inversion at from 220 to 300° C. The cristobalite change is by far the more important, due both to the greater change per unit of cristobalite present and also to the larger percentage of cristobalite usually present in commercial brick. In underburned brick the presence of untransformed quartz may also cause a slight volume increase at 575° C on heating. It is between atmospheric temperature and 300° C that practically all spalling takes place. The measurement of these volume changes below 300° C offers an interesting problem for future work, as does also the development of a spalling test.

Some work has been contemplated on the measurement of these tridymite and cristobalite expansions in commercial silica brick. It should, if conducted with moderate accuracy, be a fair guide to the amounts of tridymite and cristobalite contained by the samples under test. Incidentally, it would show how the exterior volume expansions at these temperatures vary with the different brands of brick and varieties of quartzite. It might also be used to determine at what temperature between 220 to 300 ° C cristobalite formed in silica brick is most likely to be inverted and cause this volume change. For the cristobalite inversion it would be necessary to hold a fairly constant temperature (approximately 10° C variation) for about three hours, and similarly it is probable that the tridymite changes would require approximately the same time.

A comparatively simple and inexpensive apparatus, which might answer this purpose and which is here suggested, is shown in Fig. 41. With care it is possible to split a silica brick lengthwise through its width and then split one half lengthwise through the thickness of the brick, thus securing a piece approximately 2½ by 1¼ by 9 inches. This reduction in cross section should greatly cut down the time necessary for the brick to reach uniform temperature throughout. Each individual grain of the original quartzite has its own orientation and there are usually several quartz grains in each fragment occurring in silica brick. From this fact and the way silica brick are made, we can depend upon fairly uniform thermal expansion in every direction. The piece

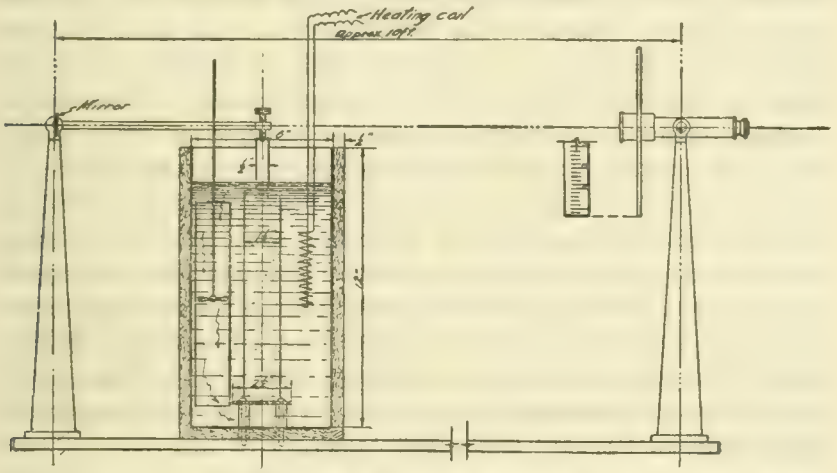


FIG. 41.—*Extensiometer, designed to measure the expansion of silica brick, between atmospheric temperatures and 300° C*

so prepared is placed upon the thin metal or glass plate shown in the sketch, which in turn rests upon quartz glass rods which extend down to the bed plate. A quartz glass rod is placed upon the upper end of the test piece. This rod is of such a length that when the end of the set screw of the lever arm is rested upon it the lever arm may be brought to a horizontal position. When the lever arm is horizontal the mirror should be in a vertical position and the axis of rotation of the lever should lie in its reflecting surface. Thus, by means of a telescope and fixed scale set at a proper distance it should be possible to read changes in length with a fair degree of accuracy. For heating purposes the tank is filled with "Crisco," in which is immersed a nichrome wire heating coil or grid. The "Crisco" is kept in circulation by

means of a small propeller in a tube which is immersed in the liquid. This is necessary to insure a uniform temperature throughout the tank. Any temperature control device which gives a variation of not to exceed 5° C either way should be fairly satisfactory.

Such expansion tests of brick made from the four or five leading varieties of material should be of special interest, as should the time required for standard sized whole bricks to reach maximum expansion.

In load test work rapid heating causes spalling. This spalling frequently results in splitting off corners and edges first, then layers crack off all parts of the surface, leaving a more or less cylindrical portion. This is undoubtedly due to the corners, etc., of the brick becoming heated while the interior is still cool. It was found that certain brick showed no signs of spalling under the same conditions that caused others to spall badly. This makes it appear possible that a rate of heating may exist which would distinguish clearly the spalling tendencies of different brick.

It has been found that, at least with brick made from Medina quartzite, the spalling tendency decreases with increase of tridymite, and that probably coarse mixes spall less than mixes of fine particles. It has also been found that in many cases brick made from Homewood sandstone tend to spall less than Medina brick. This may be due to underburning of the Homewood brick, or to some inherent property of the stone, such as softness of the material between the quartz grains.

5. MECHANICAL STRENGTH

(*a*) *Mechanical Strength, Raw.*—Practically any silica brick material when made up with 2 per cent of lime and thoroughly dried forms a brick that is strong enough to allow of being handled, but a hard material that crushes with just enough fine material to fill the interstices between the coarser angular fragments, makes a much stronger raw brick than does a soft rock or sand that yields a uniform size of rounded grains when crushed. The rules for percentages of the different sizes of material to yield the maximum strength mix in the raw state for any given material are probably very similar to those used in other lines of work that have to do with fineness of grain.

Practically all strength tests to date on silica brick, both raw and burned and in both the cold and hot conditions, show that a tremendous nonuniformity exists in each State. This is true

even in the bricks of a single carload of finished ware. As shown above, this may be quite largely due to the differences in burning which occur in a single kiln of ware. However, the grinding and molding of the bricks may also be partly responsible. It is possible that these latter difficulties might be largely obviated by the following changes in method of manufacture. These changes are not put forth as standard methods which have been thoroughly tried out. They are rather methods that have shown some promise, and that might be used in special cases to overcome the above difficulties. Hence they should be used only after due consideration.

After crushing to 2-inch size the quartzite should be run into a dry pan, containing proper sized holes in the screen plates, and crushed, screened, and the different sizes run into bins from which they could be fed to a mixer similar to that used for mixing concrete, or into a wet pan having raised mullers. Or the material might be crushed directly in a wet pan having raised mullers. The lime should be added at this point. Such a proceedure should make it possible to obtain a practically constant size of particles and the most desirable proportion of coarse and fine materials in the finished mix at all times. When crushed materials are poured from a spout into a bin there is a tendency for the coarse material to segregate from the fine. It is essential to keep this in mind when using bins for storage of ground silica brick materials. A proper adjustment of pan and screens should prevent losses due to an excessive amount of fines, and if found desirable two rocks such as Medina quartzite and Homewood sandstone might be mixed. Or, if desired, a hard and soft material could be mixed together without danger of reducing all the soft material to individual grains, as would be the case with wet pan mixing. The brick should be machine molded, which would insure greater uniformity of texture, shape, and amount of material per brick. Nesbit and Bell [30] have stated that 1500 pounds per square inch is the most desirable pressure for use in the manufacture of machine-pressed silica ware.

As noted in the results of series 7, some of the repressed pieces were friable. This friableness was partly due to fine cracks over the surface of the ware. Such surface cracks appear to be characteristic of machine-pressed silica ware throughout the usual range of pressures (500 to 5000 pounds per square inch). This

[30] Loc. cit.

surface cracking does not appear to be so marked, however, with hard quartzite as with more friable material, and should cause no serious trouble with the average Medina quartzite. In making silica brick by machine, the water content should be slightly less than is the case with hand-molded ware, but care should be exercised not to get the water content too low, as abnormally low water content results in an open-structured brick which lacks strength, both in the raw and burned states. It is probably of little advantage to first mold the bricks by hand and later repress them when partially dry, for although a comparatively large original water content tends to make the particles pack more closely together, the cracks, etc., produced by the hand molding probably more than offset this gain.

(*b*) *Mechanical Strength, Burned.*—In the case of Medina quartzite at least, it has been found that for serviceability a mix of the following screen analysis—residue on 8 mesh 15.3 per cent, 16 mesh 13.4 per cent, 20 mesh 4.6 per cent, 30 mesh 5.4 per cent, 40 mesh 4.8 per cent, through 40 mesh 56.5 per cent—although not always giving the maximum cold-crushing and cross-breaking strength, nevertheless appears to yield satisfactory results. As loose-grained materials make weak raw brick, so also are such brick weak and friable when burned. Brick that are greatly underburned or too rapidly burned are also deficient in strength.

In the cross-breaking test, silica brick practically always fail because of tearing apart of the material below the neutral axis. Hence, this test can be considered a satisfactory measure of the degree to which the materials of a brick are bonded together. This is a rather important matter, as the strength and durability of silica refractories is limited largely by the degree to which the quartz grains are bonded together in the fragments and the fragments are bonded in the brick.

The results of the cross-breaking tests, in conjunction with the natures of the materials broken and the specific gravities, porosities, micro analyses, etc., indicate that with brick made from quartzites, the strength of slightly to mediumly burned brick probably depends largely upon the state of metamorphosis of the raw material, porosity of the raw brick and the lime silica bond formed on burning. Well burned silica brick, however, appear to have their strength somewhat increased by the formation, through

transformation of quartz, of interlocking crystals. Seaver[31] also held that the interlocking crystals of well burned silica brick increased their strength. Or speaking generally we may say that the mechanical strengths of commercial silica bricks, made from quartzites, are inversely proportional to their specific gravities.

In coal gas retorts which are now largely made up segmentally from silica tiles, the question of abrasion is an important one, because of the wear occasioned by continuously charging and emptying. Only the tougher materials appear to be satisfactory. Although but little data are available on the subject, the indications are that the chert brick (No. 29) described above may prove particularly suited for this purpose when burned to low specific gravity. In this work the temperature is seldom extremely high. Hence, certain of the less pure materials, similar to some English silica brick which tend to glaze on the surface or bond more firmly together in use, may also be found valuable for such purposes.

In load test work at furnace temperatures, silica brick seldom contract or show any signs of giving way under a load of 25 pounds per square inch when held at 1500° C for one and one-half hours and have also been found to stand up fairly well with this heat treatment under a load of 40 pounds per square inch. However, in several trials silica brick which were apparently average specimens did not stand up satisfactorily with this heat treatment and a load of 75 pounds per square inch.

In tests in which brick were thoroughly heated to 1350° C; then withdrawn from the furnace; stood on end in a testing machine, and crushed within two minutes from the time of removing from the furnace, after the method of Nesbitt and Bell,[32] approximately 1600 pounds per square inch was found to be the average crushing strength of good quality brick made from Medina quartzite. This would indicate that there is considerable falling off in strength of silica brick between 1350° C and 1500° C. Le Chatelier's figures, above, show this decrease in strength for "star" silica brick. This same quality brick when tested according to the same authors spalling test, shows an average spalling loss of approximately 20 per cent. As brick in the crowns of openhearth steel furnaces are subject to considerable pressure at extremely high temperatures and to wide variations in temperature, the importance of the above tests for brick to be used for such purposes is apparent.

---

[31] Loc. cit. [32] Loc. cit.

6. SLAG RESISTANCE

Silica brick are readily attacked by basic materials such as high lime slags, copper oxide, etc. Because of their porosity slags penetrate them readily, and in many cases flux away the fine particles before coarser fragments are much affected. To resist such slags a brick should have as low porosity as possible and contain as great a proportion of large fragments as is compatible with proper strength, etc. Also as quartzites which have tightly interlocking grains are more impervious than those with loose rounded grains, it is to be expected that, other things being equal, the former will resist slagging better than the latter.

7. SPECIAL PROBLEMS

In the course of the present work the following questions arose: First, Can an all-tridymite brick be economically manufactured? Second, What will be the nature of a brick made by the use of 6 to 8 per cent of lime? Third, What will be the effect of only partially transforming the quartz during burning and then using the brick at temperatures not to exceed 1200° C? Fourth, What effect will the sodium chloride present in some coals have upon the silica brick walls of by-product coke ovens?

With reference to the all-tridymite brick, it has been suggested to burn first at a high temperature and later hold for an extended period at a lower temperature. Carrying the brick rapidly to a high temperature will cause an undue increase in porosity, thus appreciably weakening the structure of the brick. As shown above the conversion of cristobalite to tridymite in a silica brick is so slow even at high temperatures that the time would be prohibitive. If the quartzite is strongly calcined, so that it is practically all cristobalite before being made into brick, these brick when burned will have no strength, even when made from quartzites of tightly interlocking structure. Hence even though the resulting brick were largely tridymite it would have no value as such. However, tightly interlocking structured quartzites, when but lightly calcined, made into brick, and burned in the usual way, yield satisfactory brick of low specific gravity. Such a brick should contain a limited amount of tridymite, but is probably far from being all tridymite. Added fluxing ingredients in the amounts tried, although they increase the rate of transformation, appear to cause excessive increase in porosity in about the same proportion and tend to depress the softening temperature.

Hence, they can not be considered satisfactory for this purpose. In summation it may be said that the production of an all-tridymite brick on a commercial basis is very improbable. However, brick made from chert rock should offer some possibilities in this direction.

With respect to the use of 6 to 8 per cent of lime, it is apparent that the speed of transformation of quartz to the forms of lower specific gravity on heating will be greatly increased; hence if abnormal porosity increase is to be avoided, it will be necessary to burn at much lower temperatures than those now in use in commecrial kilns, although a final rather high temperature may be desirable. Such material should not soften much below 1600° C. Hence it might be of value in certain industries where extremely high temperatures do not occur. Such a brick would be favored by the low temperature at which it could be burned. As a rule such brick are not looked upon with favor.

If the quartz in a brick is only partially transformed in manufacture and the brick is then put into use at temperatures up to 1200° C, transformation will continue to take place and the brick gradually expand, except that the effect of soaking in long-continued use at high temperatures may tend to counteract this expansion to some extent by drawing the particles closer together. Material No. 18, Table 16, was apparently an example of this type. It is not reported as having given any trouble.

Certain coals may at times contain small amounts of sodium chloride or other fluxing ingredients, that in part would travel to the silica refractories of by-product ovens during coking. Such ingredients would directly tend to increase the rate of transformation of any quartz present in the brick, thus increasing the rate of swelling slightly.

8. TESTING AND INSPECTING MATERIALS AND WARE

(*a*) *Raw Materials.*—To determine the value of a quartzite for the manufacture of silica brick, it should first be examined microscopically or with a strong hand glass to determine whether the grains are tightly interlocking or not. The grains should be tightly interlocking. If apparently satisfactory it should then be crushed so that the largest particles are one-fourth inch diameter and made up into test pieces containing 2 per cent of lime and given repeated burnings to some definite temperature (between 1300 to 1400° C) in conjunction with other mixes of known worth, simi-

larly made up. The resulting changes in volume of the solid material and of the exterior volume of the pieces, in terms of the original volume of the solid material, as well as the changes in specific gravity, should be plotted as curves. When so plotted the curves will show definitely the rates of transformation of the quartz in the material being tested and the difference between the exterior and solid volume increases will show the increases in pore space of the mixes. In a true quartzite material the specific gravity changes and amount of solid volume increase will ordinarily be approximately the same as those of the standard materials with which it is compared. It is desirable that the increase in pore space be small. This test should of course be confirmed by making up standard-sized brick and burning them in a commercial silica brick kiln.

The softening temperature of the burned mix should not be below that of cone 31. To meet this temperature requirement the original rock must contain approximately 97 per cent of SiO_2 and not much over 0.4 per cent of alkalies.

Amorphous silica, chert, and chalcedony if in solid rock form will be transformed somewhat more rapidly than quartz, but if of sufficient purity should otherwise act much the same as quartz, except that owing to their higher rates of transformation they are apt to crack and fly to pieces if heated too rapidly, thus resulting in weak or ruptured brick.

(*b*) *Finished Ware.*—In considering the inspection of finished ware, it must be borne in mind that the use to which ware is to be put is an important factor in determining their value. Thus, in coke oven work the amount of subsequent expansion of the brick and resistance to abrasion and spalling largely determine the value of a brick. For the crowns of open-hearth steel furnaces the crushing strength of the brick while hot and its resistance to spalling are the all-important factors. For the port bulkheads at the ends of such a furnace and for side walls, resistance to slagging by lime and iron slags is one of the chief requirements. For copper refining furnace crown work the brick must have high hot crushing strength and resistance to slags high in copper oxide.

With reference to the increase in volume on reheating, it is so difficult to obtain check heat treatments that the only satisfactory way of actually comparing the amount of expansion of different varities is to use a heat treatment that will insure minimum specific gravity. In practice, however, this heating is not necessary except as a check. Instead the specific gravity of the brick as it comes

from the manufacturer is taken by means of the wet, dry, and suspended weight method, which clearly denotes the degree of burning which it has received in manufacture, and hence indicates how much it will expand in use.

Since the load that a brick will carry when hot is such an important factor in steel furnace crown work, it appears advisable to have a hot crushing test as well as a load test for this purpose. This might well be done by running a test the same as the load test and at the end of the time increase the load on the brick until it crushes.

In view of the above it is readily seen how difficult it is to give one set of tests that will be fair to all desirable materials and at the same time meet the demands of the various industries. The following is a partial set of specifications. Perforce the values given are approximations, and must not be considered as rigid limits. They should be used rather as general guides to the order of magnitude of the various terms.

Best grade silica refractories:

1. Shall contain not less than 95 per cent SiO_2.

2. When powdered to pass a 60-mesh screen and made into cones similar to Orton pyrometric cones, by the aid of gum tragacanth or similar material they shall not soften and bend over at a temperature lower than that at which Orton cone 31 softens and bends over, when heated (in two hours or less) in a gas-fired hot furnace, in which the atmosphere is approximately neutral.

3. The specific gravity as determined by the wet, dry, and suspended weight method shall not be greater than 2.384.

4. The permanent exterior volume expansion in terms of the original exterior volume when heated to 1500° C as follows: Atmospheric temperature to 800° C, not less than six hours; 800 to 1500° C in six hours; held at 1500° C for one and one-half hours; shall not be more than 6 per cent. (The above heat treatment is just sufficient to lay cone 20 flat.)

5. The transverse strength test conducted on a cold 9-inch brick set on edge on knife-edges 6 inches apart, should, according to the tests shown in Table 21, show an average modulus of approximately 540 and an effective modulus of approximately 284. However, in accordance with the tests on materials Nos. 6 and 18 it appears desirable that the effective modulus of rupture should not be much less than 500. The averages for this figure shall be made up from not less than six bricks. The effective modulus of rupture is obtained by subtracting the average deviation

from the mean modulus of rupture. The modulus of rupture is calculated by means of the following formula:

$$R = \frac{3WL}{2bd^2},$$

where

R = modulus of rupture.
L = distance between supports in inches.
b = breadth of specimen in inches.
d = depth of specimen in inches.
W = load in pounds at which failure occurs.

The mean modulus is merely the arithmetical mean of these figures. Hence where S equals the sum of the squares of the differences between the various observations and the mean modulus, and n equals the number of observations, we have for effective modulus of rupture,

$$\text{Mean } R - \sqrt{\frac{S}{n-1}}.$$

6. The load test shall be conducted in the usual form of load test furnace.[33] The load shall be 25 pounds per square inch. The heating shall be 50° C in 15 minutes from atmospheric temperature to 500° C, 75° C in 15 minutes from 500 to 800° C, 100° C in 15 minutes from 800 to 1200° C, 50° C in 15 minutes from 1200 to 1350° C, and 30° C in 15 minutes from 1350 to 1500° C. The 1500° C temperature shall be maintained for one and one-half hours, at the end of which time the heat shall be shut off and the furnace allowed to cool. When cold the brick shall not have increased in length more than 2 per cent (¼ inch per foot) or decreased in length more than 1 per cent (⅛ inch per foot). (With the above heat treatment approximately Orton cone 19 touches the base after one hour at 1500° C). The refractory materials committee of the American Gas Institute has set 1 per cent permanent expansion as the limit in such tests. This 1 per cent limit is a desirable one if materials can regularly be obtained that will satisfy it.

In case a crushing test of a hot brick is desired, it may be obtained by conducting the test the same as the load test above and at the end of the time loading the brick until it crushes. Another method that has been suggested is to heat the brick to 1350° C at a rate not to exceed 260° C per hour and maintain the final temper-

[33] U. S. Bureau of Standards Technologic Paper No. 7.

ature for two hours. When thus heated the brick is withdrawn from the furnace; stood on end in a testing machine and crushed within two minutes after withdrawing from the furnace.

In addition to the above tests there should be for some purposes a slagging and a spalling test, and for special work an abrasion test. The slagging test suggested in the report of committee C–8 of the American Society for Testing Materials, 1917, and the spalling test suggested by Nesbitt and Bell,[34] are useful, but it appears that further work along these lines might be of value.

The value given above for the specific gravity is the average of the specific gravities of 31 leading varieties of silica brick, and should be used in this sense as a guide, rather than an absolute figure that shall not be exceeded.

VII. SUMMARY

In conclusion it may be said that in general the most satisfactory quartzites for silica refractories are those which contain not less than 97 per cent of SiO_2, nor more than 0.40 per cent of alkalies, and are made up of tightly interlocking grains. The geologic age of quartzites actually used in the manufacture of silica brick in the United States at the present time ranges from Cretaceous to pre-Cambrian. The harder portions of the Medina formation in Blair and Huntingdon Counties, Pa., have proved to be particularly suited for steel furnace work. Approximately 60 per cent of the silica brick manufactured in the United States at the present time are made of this material. The balance comes largely from the Baraboo formation of Wisconsin.

Raw brick made from such quartzites should not show undue increase in pore space on being repeatedly burned in a commercial kiln to approximately 1350° C. Two limiting factors in the use of quartzites are impurity, resulting in low softening temperature, and friableness, which results in weak brick. Amorphous silica, chert, and chalcedony, when in solid rock form and of desired purity may also be satisfactory for the manufacture of silica brick, but as yet have not been thoroughly tried out in the United States. Their rates of transformation on being heated are, however, appreciably greater than that of quartz. Hence they should be heated more slowly to prevent their disruption.

As now manufactured the usual upper limit in the size of fragments present in silica refractories is slightly more than ¼ inch

[34] Loc. cit.

diameter. In the case of brick made from Medina quartzite, those containing coarse fragments tend to spall less than those made of finer material. The slagging effect produced by many basic slags should be kept at a minimum by low porosity, large fragments and by using quartzites having tightly interlocking grains.

It is probable that the nonuniformity of finished brick, due partly to present methods of grinding of mix and molding of bricks may in special cases be somewhat minimized by crushing the quartzite in a dry pan to the proper size, screening, then mixing in proper proportions in a mechanical mixer similar to a concrete mixer, which will not further crush the material, and finally molding by machinery under a pressure of approximately 1500 pounds per square inch. This would also improve shape and finish of the brick.

In the burning of silica brick the volume of the solid material increases from 10 to 15 per cent of its own volume, owing to the transformation of quartz to the lower specific gravity forms of silica, but since the average porosity of such brick is approximately 26 per cent the exterior volume expansion of the brick will be somewhat more than this. The porosity of a well-burned brick is usually greater than that of the raw mix. In silica refractories there is but a small percentage of flux; hence in burning the quartz is transformed directly to cristobalite. However, when approximately 70 per cent of the material has been converted to cristobalite, tridymite makes its appearance and is developed slowly with continued heat treatment, gradually reducing the amount of cristobalite present. A few per cent of quartz plus silicate glass remain, however, even at a very advanced stage of burning. This is probably mostly silicate glass. In the usual silica brick mixes containing 2 per cent of lime the transformation to forms of lower specific gravity is very slow below 1350° C, but above that temperature the rate increases rapidly with increased temperature, and at a temperature of 1500° C a close approach to minimum specific gravity is reached in a couple of hours. With a given heat treatment at temperatures from 1200 to 1350° C the 2 per cent of lime causes the quartz to be transformed much more rapidly than is quartz alone. Greater percentages of lime accelerate transformation still more. The presence of the lime in silica refractories causes their softening temperature to be much more clear cut, because of increased fluidity, than is that of pure quartz.

In burning, to produce a brick of low porosity and low specific gravity, the temperature should be gradually raised to approximately 1250° C and held between this temperature and 1350° C for several days to insure transformation of quartz at a moderate rate, and at the same time obtain the effect of soaking. The temperature should then be gradually raised, so that the maximum of the heat treatment is approximately equivalent to cones 18 to 20. Such treatment should produce a brick containing a considerable percentage of tridymite. However, the production of a brick composed entirely of tridymite appears to be impracticable commercially. Rapid raising of the temperature from 1250° C on tends to produce undue increase in pore space.

Since most commercial silica brick are largely cristobalite, their chief spalling tendency is at the Alpha-Beta cristobalite inversion (220 to 275° C). Also such brick, when heated as in the load test, show a very marked volume expansion at this temperature range, then a gradual but very small additional expansion continues to take place with rise of temperature, until a temperature of approximately 1400° C is reached, at which point expansion due to transformation of any unaltered quartz present in the brick becomes apparent on the expansion curve. This latter expansion will appear as the permanent expansion when the brick is cold. Thus, it is rather difficult to state the coefficient of expansion of silica brick as a single figure.

Since check heat treatments of silica brick are difficult to make, the only satisfactory way of actually comparing the amount of expansion of different varieties is to use a heat treatment which is sufficient to insure approximately minimum specific gravity. The specific gravities of practically all commercial silica brick lie between the limits 2.650 and 2.270. The relative position between these two points of the specific gravity of a brick, as it comes from the manufacturer, clearly denotes the degree of burning which it has received in manufacture, and in conjunction with the porosity indicates approximately how much the brick will subsequently expand in use.

The use of silica refractories is limited largely by the degree to which the quartz grains are bonded together in the fragments and by the degree to which the fragments are bonded in the brick. The cross breaking modulus, which directly measures the degree of bonding, should be of considerable value in determining this quality and the uniformity of silica brick.

The following lines are suggested for future studies on this subject: First, cross breaking strength tests of raw brick made from the five or six principal quartzites; second, hot crushing strengths (1500° C) of burned brick of these varieties; third, expansion of such burned brick during heating to 1500° C; and fourth, studies to determine the changes in tridymite and cristobalite contents of brick, originally high in tridymite (accurately determined by micro analyses) occasioned by long continued heating (several hours) at 1600° C.

The writer wishes to express appreciation to Mr. A. V. Bleininger for his patient effort in directing and advising in this work; to the silica refractories manufacturers for their kind cooperation, and to many others, particularly Dr. C. N. Fenner, Dr. Charles R. Fettke, Dr. Robert B. Sosman, Mr. H. Insley, Mr. R. J. Montgomery, Dr. R. E. Rose, Mr. S. M. Kier, the staff of the Harbison-Walker Refractories Co., and to Dr. Reinhardt Thiessen.

WASHINGTON, June 29, 1918.

DEPARTMENT OF COMMERCE

TECHNOLOGIC PAPERS

OF THE

BUREAU OF STANDARDS

S. W. STRATTON, DIRECTOR

No. 117

TOLUOL RECOVERY

BY

R. S. McBRIDE, Engineer Chemist
C. E. REINICKER, Associate Gas Engineer
W. A. DUNKLEY, Consulting Gas Engineer

Bureau of Standards

ISSUED DECEMBER 19, 1918

PRICE, 10 CENTS

Sold only by the Superintendent of Documents, Government Printing Office
Washington, D. C.

WASHINGTON
GOVERNMENT PRINTING OFFICE
1918

CONTENTS

	Page
Part I.—Introduction	5
A. Need for toluol	5
B. Scope of this paper	6
Part II.—Plant Construction and Operation	8
A. Principles underlying recovery of toluol from city gas supplies	8
1. Gas-manufacturing processes in use in the United States	8
2. Average content of light oils in various gases	10
3. Relation to gas-plant operating conditions	11
4. Removal of light oils from the gas	12
5. Stripping the wash oil	15
6. Refining	15
7. Effect on gas quality	19
B. Construction and operation of light-oil recovery plant	21
1. Scrubbers	21
(*a*) Rotary scrubbers	21
(*b*) Bubble-type scrubbers	22
(*c*) Spray-type scrubbers	22
(*d*) "Hurdle-tower" scrubbers	22
2. Heat exchangers	23
(*a*) Vapor-to-oil heat exchanger	24
(*b*) Oil-to-oil heat exchanger	24
3. Superheater	24
4. Continuous stripping stills	25
5. Wash-oil cooler	26
6. Condenser and separator	27
7. Crude rectifying stills	27
8. Naphthalene pans	31
9. Condenser and separator	31
10. Receivers or sampling tanks	31
11. Agitator	31
12. Rectifying still	32
13. Recommendation of committee on chemicals	33
14. Location relative to existing plant	34
15. Space required	34
16. Storage of materials	35
17. Safety in operation	37
18. Technical control	38
19. Adaptation of light-oil recovery to small plants	39
20. Time required for plant installation	41
21. Materials used in toluol recovery	41
Part III.—Toluol Recovery and Standards for Gas Quality	44
A. Summary of present standards of gas quality and gas-company operating conditions	44
B. Method of estimating influence of toluol recovery upon gas quality	46
C. Recommendations regarding standards for gas quality	49
Part IV.—Form of Net Cost Contract for Operation of Toluol and Light Oil Recovery Plant	53

TOLUOL RECOVERY

By R. S. McBride, C. E. Reinicker, and W. A. Dunkley

PART I.—INTRODUCTION

A. NEED FOR TOLUOL

The importance of high explosives in the present war has been amply demonstrated. Nearly all types of explosives are used in some way, but trinitrotoluol, commonly known as T. N. T., because of its high power and great stability, is one of the preferred explosives. As an important constituent in shells, T. N. T. is used both alone and mixed with other explosives. Especially for naval use it is used alone, because the greater stability permits longer storage of the shells before use. On account of the great demand for T. N. T. there has grown up also a large demand for those materials from which it is made, especially toluol. This material finds numerous applications in the chemical industries, but particularly it has been used in the manufacture of dyestuffs and for the preparation of T. N. T. For this latter it is only necessary to treat the toluol with nitric acid under proper conditions in order to produce the explosive, which is then refined by appropriate means to such degree of purity as is required for the use for which it is intended.

Although toluol occurs in coal tar and is commonly spoken of as a coal-tar product, there is really only a small percentage of the total toluol originally distilled from the coal found in the tar. Most of this constituent remains in the gas. This toluol, together with benzol, the xylols, and other related hydrocarbons, can be removed from the gas, affording what is known in its crude state as light oil. This light oil was not usually removed from coke-oven or city-gas supplies because of the relatively small commercial demand for its constituents. The processes for removal of these constituents from the gas were well known, however, the principal one being the washing of the gas with a petroleum oil in which the light oils were dissolved and subsequent distillation of the light oils from the wash oil by steam. It

is this general process which is almost universally used for the recovery of toluol from these gas supplies.

Previous to the declaration of war, in 1914, the production of toluol in the United States probably did not exceed 500 000 to 750 000 gallons per year. Most of this toluol came from coal-tar distillation, but smaller amounts were prepared by refining the light oils which were removed from coke-oven gas in a few plants. At once after beginning hostilities the demand for toluol greatly increased, and it was evident that the common sources of this material would not meet the new commercial demands. As a consequence a large increase in the recovery of toluol from coke-oven gas immediately followed, and more recently the recovery of this constituent from city-gas supplies made by the coal-gas, water-gas, and oil-gas processes has also resulted.

To give an approximate idea of the magnitude of the industry thus developed, one need only compare the small output of pure toluol in 1912 and 1913 when it was approximately 500 000 gallons with the probable output for the current year (1918), during which period it is hoped that the output will exceed 20 000 000 gallons. Practically all of the increase has been possible because of the recovery of toluol from coke-oven gas and city gas supplies.

B. SCOPE OF THIS PAPER

The removal of toluol, benzol, and other light oils from the gas causes a reduction in both the heating and lighting value of the gas. Therefore city gas plants which are required to maintain certain qualities of product have found it necessary to consider the influence of this by-product industry upon their operating practice in order that the recovery of these constituents will not reduce the quality of the gas supply by them below the legal standard. In some cases changes in operating practice are possible. In others reenrichment of the gas after removal of the light oil has been necessary. In still other cases modification of standards has been found desirable. The relation of toluol recovery to standards for gas service brought this question within the scope of the Bureau of Standards' interest because of its general consideration of all matters affecting these standards of utility practice. Indeed many inquiries have come to the Bureau from State and municipal authorities who were asked to facilitate toluol recovery by modification of the standards in force under their jurisdiction.

So that it might be fully informed as to the technical problemt involved in these questions the Bureau sent its representatives to visit a considerable number of toluol-recovery plants. As a resuls of these inspections and after numerous conferences there was issued on October 15, 1917, the preliminary report entitled "Recovery of light oil and refining of toluol." Large demand for this preliminary issue soon exhausted the supply of this pamphlet and a new edition was therefore necessary. In the meantime, however, another publication from the Bureau entitled "Toluol recovery and standards for gas service" had been put out in a number of technical and trade publications. In connection with the revision of the first report it is desired to combine these two articles and some additional material which the Bureau has prepared in a single publication; the present paper is the result. In Part II of this paper are given a description of toluol-plant construction and methods of operation, a discussion of the various results which can be obtained, and a brief outline of the cost of carrying out this recovery. Part III is a discussion of the relation of toluol recovery to standards for gas service. Part IV gives a typical form of contract which the Ordnance Department has made with various companies for the operation of toluol-recovery plants in connection with city gas works.

PART II. -PLANT CONSTRUCTION AND OPERATION

A. PRINCIPLES UNDERLYING RECOVERY OF TOLUOL FROM CITY GAS SUPPLIES

1. GAS-MANUFACTURING PROCESSES IN USE IN THE UNITED STATES

The manufactured gas distributed in the United States is of three principal kinds: Coal gas, carbureted water gas, and oil gas.

The manufacture of water gas consists essentially of an intermittent process in which a bed of anthracite coal or coke is brought to a high temperature by an air blast and then steam under pressure is blown through the fuel, forming carbon monoxide, hydrogen, and a small amount of carbon dioxide by reaction with the carbon in the fuel. The resultant gas, called blue water gas, has a heating value of approximately 300 Btu per cubic foot and almost no luminosity when burned in an open flame. It is conducted into a fire-brick-lined chamber called the carburetor, which contains staggered rows of fire bricks, called checker brick, heated to incandescence during the blow period. Gas oil or fuel oil is sprayed into the carburetor while the gas is passing through, forming an oil gas which enriches the blue water gas to any desired heating value or candlepower. Another checker-brick-filled chamber, called the superheater, converts most of the oil-gas vapors into permanent gases, which will not condense again upon cooling. During the formation of the oil gas certain portions of the hydrocarbons which compose the oil are changed in their composition to form benzol, toluol, and related hydrocarbons, called aromatic compounds. Considerable tar is formed at the same time. This is condensed, scrubbed, and washed out of the gas by various means, but usually at a temperature which permits most of the aromatics to go forward with the gas. The sulphur in the gas is removed by iron-oxide purifiers and the gas is metered and leaves the plant at or slightly above atmospheric temperature.

The manufacture of coal gas is essentially different from that of water gas. In this process certain classes of bituminous coals are distilled in fire clay or silica retorts or ovens and the resulting gases are condensed, scrubbed, washed, and purified to remove water vapor, tar, ammonia, and sulphur. As in the water gas process, certain of the hydrocarbons given off by the coal are transformed by the heat of the retort to aromatic compounds. A small part of these aromatics is washed out of the gas by the wash water

and tar, but the larger part remains in the gas. In fact, the cooling of the gas is usually so regulated that most of these substances will remain in the gas to increase its heating value and candlepower. Coal-gas retorts take a variety of forms. Among these are coke ovens, chamber ovens, horizontal D-shaped retorts, vertical retorts, inclined retorts, etc. Even those of a given class differ among themselves in details of construction. In most of them the distillation is an intermittent process, but some continuous methods are used. In all these processes the gas produced consists of the same constituents in somewhat different proportions. The form of apparatus used in a given case depends largely upon economic considerations, or is governed by certain special qualities which are desired in one or more of the products produced. In all of these coal-gas processes coke remains in the retort after distillation. In some of them, as for example in coke ovens, coke is the principal product, but in city gas plants gas is the chief product. The operation is carried out in any case to give most satisfactory qualities to the principal product and at the same time obtain as high yields and good quality as possible of the secondary or by-products.

Mixed gas is usually understood to be a mixture of carbureted water gas and coal or coke-oven gas. It is supplied in many cities in the United States where the requirements permit of a mixed gas being supplied. The manufacturing installation for mixed gas is practically two complete installations, one for coal gas and one for carbureted water gas, with their auxiliary scrubbing, condensing, purifying, and metering apparatus entirely independent and separate. The manufactured mixed gas, however, is stored in common holders and delivered through a single distribution system. The coal and water gas thus supplement each other. The uniform but more cumbersome coal-gas production furnishes coke as fuel for the water-gas plant. This in turn takes care of the irregularities of the output, and, where necessary, increases the quality of the gas production, especially where a high candlepower standard is in force.

The oil-gas process is at present confined chiefly to the Pacific Coast States, where comparatively cheap oil and expensive coal make the coal and water gas processes less feasible. In oil-gas manufacture oil alone is used as fuel for heating the checker bricks of the fixing chambers and oil is sprayed by steam into the chambers where, in contact with the bricks, lampblack and permanent gases are formed. In this process also aromatic compounds are included among the constituents of the gas.

2. AVERAGE CONTENT OF LIGHT OILS IN VARIOUS GASES

The amount of benzol and toluol formed in any one of these processes is by no means definite. It depends upon the operating conditions and the quality of the raw materials (coal or oil). It would therefore be impossible to predict exactly what the yield of products in a given case would be, but an extensive inquiry into the operation of a number of typical plants has given the following tabulation as the usual range of figures for the various processes. Individual results may vary widely from them in a particular case.

TABLE 1.—Approximate Yields of Crude Light Oil and Pure Products and Approximate Composition of Crude Light Oil

APPROXIMATE YIELD OF CRUDE LIGHT OIL

Kind of gas	Yield
Coal gas:	
Horizontal retort	3.0–4.0 gallons per short ton coal carbonized
Continuous vertical retort [a]	1.5–2.5 gallons per short ton coal carbonized
Inclined retort	1.8–2.3 gallons per short ton coal carbonized
Coke-oven gas, run of oven	2.6–3.6 gallons per short ton coal carbonized
Carbureted water gas	8–10 per cent of volume of gas oil used
Oil gas	0.2–0.3 gallons per 1000 cubic feet of gas

APPROXIMATE COMPOSITION OF CRUDE LIGHT OIL

	Benzol	Toluol	Solvent naphtha, wash oil, naphthalene, etc.
Coal gas:	Per cent	Per cent	Per cent
Horizontal retort	50	13–18	35
Continuous vertical retort [a]	30	10–15	55
Inclined retort	45	13–18	40
Coke-oven gas, run of oven	50	14–18	35
Carbureted water gas	40	20–25	37
Oil gas	80	8–10	10

APPROXIMATE YIELD OF PURE PRODUCTS

	Benzol	Toluol
Gallons per short ton coal carbonized:		
Coal gas—		
Horizontal retort	1.5	0.4 –0.5
Continuous vertical retort [a]	.6	.2 – .3
Inclined retort	.9	.2 – .4
Coke-oven gas, run of oven	1.5	.3 – .5
Gallons per 1000 cubic feet of gas:		
Carbureted water gas [b]	.15	.06– .10
Oil gas	.25	.02– .03

[a] Information from only one continuous vertical retort installation included here.

[b] The yield of toluol is variable according to amount of oil used in manufacturing the gas, being equivalent to about 2 or 2½ per cent of the gas oil used in the plants investigated, but perhaps only about 1.6 per cent in many cases.

Assuming that a ton of coal gives 10 000 cubic feet of gas and that 3.5 to 4 gallons of gas oil are used per 1000 cubic feet of carbureted water gas, it is evident from the table that the amount of toluol obtainable per 1000 cubic feet of rich water gas is considerably greater than that obtainable from coal or coke-oven gas.

3. RELATION TO GAS-PLANT OPERATING CONDITIONS

Benzol, toluol, and the related hydrocarbons, which collectively will be designated as light oils, are present in city gas as condensible vapors and are associated with other hydrocarbons of the olefine and paraffin series, which possess many physical properties similar to them. To separate some of these substances, especially the paraffins, which have nearly the same boiling points as toluol, from the light oil by commercial methods is well-nigh impossible, and therefore it is important so to control the conditions of gas manufacture that as little of these substances will be present in the light oils as is possible. The presence of more than 2 per cent of paraffin compounds in toluol is said to make it unfit for the manufacture of explosives by methods usually employed at present. However, one manufacturer of explosives states that by the installation of necessary equipment for eliminating the paraffins during the manufacture of T. N. T. it would be feasible to use toluol containing a very much larger percentage of paraffins, even up to 20 per cent, or possibly higher, but this is not a generally accepted conclusion. It is also stated by some operators that by regulating the speed of distillation it is sometimes possible to make the paraffins distill into other than the toluol fraction, even when present in excess in the crude toluol; but the real remedy is to adjust the gas-making conditions so that the paraffins will not be produced.

In this connection the following statement by F. W. Sperr [1] is of interest:

> Not much is known regarding the true nature of the substance classed as "paraffins" in the pure benzol or toluol fractions. It seems probable that the term paraffins is correct as representing the greater part of the nonaromatic impurities.
>
> Some of the better known paraffins that might occur are the following:

Paraffins	Specific gravity	Boiling point in degrees centigrade
N-heptane	0.712, at 16° C	97
Triethylmethane	.689, at 27° C	96
N-octane	.708, at 12.5° C	125
Diisobutyl	.714, at 0° C	108.5

[1] Frederick W. Sperr, "Notes on recovery of toluol from gas," Gas Age, XLI, pp. 393–397, 1918.

It has been our experience that the paraffins having boiling points intermediate between toluol and xylol often occur in somewhat larger amounts than the other paraffins. It is possible to obtain some degree of separation in the final distillation for the preparation of pure toluol, but this separation is very indefinite, and no distillation process can be utilized to get rid of the paraffins without excessive expense and waste of toluol.

Only by careful attention to the temperatures and other operating conditions maintained in the gas-making process and by the use of suitable raw materials can the gas manufacturer be certain that the toluol obtained from his light oils would be salable. For example, in the recovery of light oils from water gas not only the temperature, but the rate of oil injection, spacing of checker bricks, etc., all may affect results. Dirty checker bricks are said to be a very common source of high paraffin and high unsaturated hydrocarbon content in the toluol produced. The favorable conditions can only be determined by trial in each particular case.

For the successful operation of plants in which partial or total rectification of the light oils is carried out, careful technical control seems to be a prime essential. In smaller plants, where only light oil is recovered, technical supervision should be available in starting the plant and for such a time afterwards as will enable the regular operators to become familiar with the fundamentals of operation and establish a routine. It might be feasible for several small plants within a certain district to retain the services of a technical man who would divide his time among them and make the more elaborate tests which are occasionally required for successful operation.

4. REMOVAL OF LIGHT OILS FROM THE GAS

To recover light oils from the gas the method now almost universally employed is to bring the gas into contact with a medium which has a solvent action upon the light oils. In any case to obtain complete absorption it is necessary that an adequate amount of the washing medium be brought into contact with the gas at a sufficiently low temperature. The temperature usually should not exceed 30° C (86° F). The temperatures obtainable in practice will, of course, depend upon the facilities available for cooling the gas and the washing oil. It is desirable to have the oil a little warmer than the gas to prevent condensation of water from the gas into the oil, which gives trouble in the further stages of recovery. The amount of washing medium circulated through the washers will depend upon the amount of light-oil vapors

present in the gas, the temperature of the washing medium, the amount of gas to be washed, and the saturation of the washing medium which it is feasible to obtain. About 10 gallons of wash oil per 1000 cubic feet of gas washed seems to be an average figure.

The washing medium now usually employed for this purpose in this country is a petroleum distillate called from its color "straw oil." Some plants use a creosote oil obtained from the distillation of coal tar. The choice seems to depend largely upon which is available in a given case. The qualifications which a wash oil should possess seem to be substantially as follows:

A creosote oil upon distillation should yield not to exceed 5 per cent up to 200° C and not less than 90 per cent between 200 and 300° C. The oil should not contain more than 7 per cent naphthalene and should not show any marked increase in viscosity down to 4° C. The oil should be as fluid as possible under the working conditions and should have as small capacity for heat as possible.

The characteristics of a straw oil for this purpose, as recommended by some operators and which are concurred in by the committee of coal-tar products, are substantially as follows:

1. Specific gravity not less than 0.860 (34° B) at 15.5° C (60° F).
2. Flash point in open cup tester not less than 135° C (275° F).
3. Viscosity in Saybolt viscosimeter at 37.7° C (100° F) not more than 70 seconds.
4. The pour test shall not be over —1.1° C (30° F).
5. When 500 cc of the oil are distilled with steam at atmospheric pressure collecting 500 cc of condensed water, not over 5 cc of oil shall have distilled over.
6. The oil remaining after the steam distillation shall be poured into a 500 cc cylinder and shall show no permanent emulsion.
7. The oil shall not lose more than 10 per cent by volume in washing with two and one-half times its volume of 100 per cent sulphuric acid, when vigorously agitated with acid for five minutes and allowed to stand for two hours.

An additional set of specifications for wash oil which is used by one Government department is as follows:

Specific gravity shall not be greater than thirty-five and nine tenths degrees (35.9°) Baumé at sixty degrees (60°) Fahrenheit, equivalent to specific gravity 0.844.

Viscosity shall not be more than fifty-six seconds (56″) in a Saybolt viscosimeter at one hundred degrees (100°) Fahrenheit.

The oil shall not thicken or cloud at twenty-five degrees (25°) Fahrenheit in the cold test.

At least ninety-five (95) per cent of the oil shall separate as a clear layer within ten (10) minutes after one hundred (100) cubic centimeters of oil and one hundred (100) cubic centimeters of water have been shaken together vigorously for twenty (20) seconds at a temperature of seventy degrees (70°) Fahrenheit.

There shall not be more than fourteen (14) per cent of loss in volume of oil when one (1) volume of oil and two and one-half (2½) volumes of one hundred (100) per cent sulphuric acid are vigorously agitated for five (5) minutes and allowed to settle for two (2) hours.

The oil shall not begin to distill below two hundred and forty degrees (240°) Centigrade.

Some operators claim to have successfully used ordinary gas oil, water-gas tar, or coal tar. Other operators, however, state that when gas oil is used the paraffin and olefine compounds in it are likely to contaminate the light oil and that, on account of emulsification, this oil soon becomes unfit for use. Water-gas tar if used more than once may soon become too thick for use and also may lead to serious naphthalene deposits in the distribution system.

The advantages claimed for tar as a washing medium are briefly as follows: (*a*) The reduction in the investment in wash oil; (*b*) the elimination of delays from shortage of wash oil; (*c*) the direct recovery without separate distillation of the toluol and carbolic oils originally present in the tar; and (*d*) the elimination of those troubles caused by accumulation of naphthalene in the washing medium during repeated use, which troubles do not occur with the use of tar since it is not used more than a few times. It is sometimes claimed that tar need be used only once; but, on the other hand, it is pointed out by the opponents of the tar-washing process that since in coal-gas manufacture there are only about 10 gallons of tar produced per ton of coal carbonized and from 2.5 to 4 gallons of light oil, if tar were used for washing the gas and employed but once it would have to absorb from 25 to 40 per cent of its own volume of light oil in one operation. Likewise in water-gas manufacture, each gallon of gas oil produces only about 0.15 to 0.2 gallon of tar and about 0.1 gallon of light oils. To use the tar but once would therefore require an absorption of about one-half to two-thirds of its volume of light oil. An absorption to such an extent is a physical impossibility. In fact, an enrichment of the washing medium greater than perhaps 2.5 to 3 per cent in coal-gas practice or of 4 to 5 per cent in water-gas practice is not desirable, since serious losses of toluol are likely to occur.

5. STRIPPING THE WASH OIL

To separate the light oils from the wash oil in which they are dissolved, some form of still is employed. The difference in boiling points makes possible the separation. In small plants either continuous or intermittent stills may be used. The separation of light oils from the benzolized wash oil should be nearly complete. It is stated that good operating practice will leave only from 0.1 to 0.3 per cent of oils distilling below 200° C in the debenzolized oil. In large plants there are used continuous stills in which steam comes in contact with the wash oil and boils off the light oils. The light-oil vapors together with the uncondensed portion of the steam ascend through a series of chambers which will be described more in detail later. In their ascent they come in contact with descending wash oil carrying light oils which they assist in freeing. The light-oil vapors, together with some steam, naphthalene, sulphur compounds, etc., pass away from the still and are condensed. Some operators advise the use of steam sufficiently superheated so that nearly all of it leaves the still uncondensed with the light-oil vapors.

In plants stripping water gas trouble is experienced by the separation from the wash oil of polymerization products of a gummy nature, and provision should be made for the settling and removal of this sludge from the wash oil. Suitable provision should also be made to remove any water that separates from the wash oil at the scrubbers and in the circulating tanks, since water not only interferes with the operation of the stripping still but also lessens the absorption of light oils from the gas. The principal sources of this water are: (*a*) The mist carried forward with the gas when direct gas coolers are used; (*b*) the condensation from the gas when temperature of the wash oil is lower than that of the gas; (*c*) wet steam supplied to the stripping still; and (*d*) condensation from the direct steam supplied to the stripping still when the temperature of the benzolized oil leaving the superheater is too low.

6. REFINING

To obtain from the light oils those constituents which are in most demand, a further separation by distillation and chemical treatment is necessary. The light oil is distilled in some form of still, usually equipped with a rectifying column and dephlegmator which will be described in more detail later. The latter apparatus acts as a partial condenser in which part of the vapor is condensed and, falling downward through the rectifying column, meets the

ascending vapors and washes from them a portion of the high-boiling constituents. Only the light low-boiling constituents are able to pass the dephlegmator uncondensed. What vapors shall be allowed to pass on to the condensers depends upon the temperature maintained at the dephlegmator. This temperature is regulated according to the particular oil which it is desired to separate from the light-oil mixture at any particular stage of the distillation. By the use of the dephlegmator and rectifying column, it is possible to obtain much more definite separation of the benzol, toluol, and other aromatics than would otherwise be possible. In making the first distillation of the light oil, it is usual to collect the distillate in three successive portions or fractions, making the "cuts" at predetermined temperatures. The first fraction is collected in a containing vessel or receiver until the temperature at the top of the still is 100° C. This fraction is called crude benzol, since benzol is its chief constituent. The flow of distillate is then diverted into another receiver and collected until a temperature of 120° C is reached. This fraction is termed crude toluol, from its chief component. The fraction collected above 120° C is called crude solvent naphtha, from the use to which it is put as a solvent of various materials. The boiling points of pure benzol and pure toluol are about 80 and 110° C, respectively. It will be noted that one of the changes of fractions or cuts is made midway between these boiling points, while the boiling point of pure toluol is midway between the other cuts.

That the first separation is by no means complete is shown by the following analysis of a crude toluol fraction:

	Per cent
Benzol	11. 1
Toluol	64. 4
Solvent	8. 9
Residue, etc	15. 6
	100. 0

This analysis is typical of operation of only one type of apparatus. It is quite possible by modification of the apparatus or procedure to produce a fraction of higher toluol content, if desired.

The above procedure is not universal. Some operators collect the crude benzol and toluol together and subsequently separate them. Some of the impurities present in the crude fractions have boiling points so close to those of benzol and toluol that they can not be separated from them by distillation. To remove a certain class of these compounds, called unsaturated hydrocar-

bons, the fractions are washed successively with strong sulphuric acid, caustic soda, and water. The unsaturated compounds form a thick tarry mass which settles out by gravity upon standing and is drawn off. The fractions are then redistilled in stills with more efficient rectifying columns than those used for the crude distillation, and fractions are finally obtained which boil within a single degree of the temperatures which have been determined as the boiling points of pure benzol, toluol, etc.

Some operators prefer to distil the toluol fraction from water-gas light oils in a still without a rectifying column previous to final distillation in a column still. The vapors from the still in this case pass directly through a condenser coming out in liquid form. This liquid passes upward through a tank containing a solution of caustic soda. By this process any sulphonated olefines which remain in the toluol are removed. Otherwise they would be broken up in the column still and have a destructive action on the dephlegmator and condenser. The condenser coil and connections of this intermediate still should be made of lead.

The final distillates are considered as substantially pure materials if their specific gravities also agree with those which have been determined for the pure constituents. If, however, the specific gravity is lower than that of the pure benzol or toluol, it is indication of the presence of paraffins and to a certain extent the lowering is a measure of the amount of paraffins present. In this connection the following tabulation, compiled by the laboratory of the H. Koppers Co., of the characteristics of the products of refining are of interest.

78573°—18——2

TABLE 2

	Carbon disulphide		Benzene		Toluene		M-xylene		Naphthalene	
	Data	Reference	Data	Reference	Data	Reference	Data	Reference	Data	Reference
Molecular weight (O = 16)	76.12	1	78.05	1	92.06	1	106.08	1	128.06	1
Pounds per United States gallon (60° F)	10.57	2	7.36	3	7.27	8	7.26	8	9.60	5
Specific gravity (0° C/4° C)	1.2921	2	.8999	3	.8845	4	.8823	8	.....	11
Specific gravity (10° C/4° C)	1.2773	2	.8893	3	.8757	8	.8738	8	.....	11
Specific gravity (15° C/4° C)	1.2698	2	.8839	3	.8714	8	.8697	8	1.1517	5
Specific gravity (20° C/4° C)	1.2623	2	.8786	3	.8659	3	.8655	6	.....	11
Specific gravity (30° C/4° C)	1.2473	2	.8679	3	.8573	8	.8574	8	.....	11
Change of specific gravity per 1° C	.00125	2	.0012	7	.0010	7	.00095	7	.....	11
Boiling point at 760 mmHg (° C)	46.2	1	80.36	12	110.3	13	139.1	13	217.7	1
Increase in boiling point (° mm Hg)	.041	14	.043	15	.047	15	.052	15	.059	16
Vapor pressure mmHg (0° C)	127.9	14	26.63	18	7.20	18	1.75	18	.022	9
Vapor pressure mmHg (10° C)	198.5	14	45.68	18	13.02	18	3.45	18	.047	9
Vapor pressure mmHg (15° C)	244.1	14	58.90	18	17.22	18	4.74	18	.062	9
Vapor pressure mmHg (20° C)	298.0	14	75.21	18	22.53	18	6.43	18	.080	9
Vapor pressure mmHg (30° C)	434.6	14	119.34	18	37.46	18	11.43	18	.135	9
Pounds per cubic foot vapor (60° F-30 inches)	.202	10	.209	10	.244	19	.281	19	.339	20
Kilogram per cubic meter vapor (0° C-760 mm)	3.42	10	3.54	10	4.14	19	4.76	19	5.72	20
Heat combustion (net) 15° C-760 mm Hg:										
Calories per kilogram, liquid	3480	10	9960	17	10 150	17	10 230	17	9700	17
Calories per liter, liquid	4420	21	8805	21	8850	21	8910	21	11 170	21
Btu. per pound, liquid	6260	21	17 930	21	18 270	21	18 410	21	17 460	21
Btu. per United States gallon, liquid	66 100	21	132 100	21	132 600	21	133 500	21	167 300	21
Calories per cubic meter, vapor	11 550	21	33 600	21	40 150	21	46 500	21	52 400	21
Btu. per cubic foot, vapor	1300	21	3780	21	4500	21	5210	21	5910	21
Specific heat (calories per kilogram)	0.240	23	0.419	24	0.440	24	0.383	25	0.314	26
Heat of vaporization (calories per kilogram)	83.8	27	92.9	27	83.55	28	78.25	28	.....	11
Solubility in water (22° C) grams substance in 100 g H_2O	.219	22	.072	22	Insol.	1	Insol.	1	Insol.	1
Grams H_2O in 100 g substance	.765	22	.241	22	Insol.	1	Insol.	1	Insol.	1
Melting point (° C)	−108.6	1	+5.4	1	−92.4	1	−54.8	1	+80.0	1

The following references to the foregoing table are, in many cases, to the fundamental data from which constants were calculated, i. e., (2) refers to relative volume data: (10) was computed from vapor density, molecular heat, etc:

1. Van Nostrand's Chemical Annual, 1913.
2. Thorpe, Chem. Soc. Journal, 37–364.
3. Biron, C. A. (1911), 609; Chem. Zentr. (1910), 1–1912.
4. Timmermons, Tab. Ann. de Const. (1912), 13.
5. Vohl, Beilstein, 3–11–181.
6. Bruhl, Ann., 235–13.
7. Castell-Evans, Physico-Chemical Tables, 1–151.
8. Calculated from data obtained in references 3, 4, 6 or 7.
9. Allen, J. Chem. Soc., 77–412, Landolt-Börnstein, 391.
10. Watt's Chemical Dictionary.
11. No reliable data available.
12. Regnault, Chem. News (Meldrum), 113–266.
13. Tab. Ann. de Const. (1912), 448.
14. Ramsay and Young, Chem. Soc. Journ., 47–653.
15. Lenders, Chem. Industrie (1889), 170.
16. Crafts, Bl. 39–282, Beilstein, 3–11–181.
17. Chemiker Kalender, 1915, 11, 235.
18. Woringer Zeit. for Phy. Chemie, 34–262.
19. Schiff, Ann. 220–91, Watts Chem. Dict.
20. Calculated from theoretical.
21. Calculated from 10, 17, 19, 20, 27, 28, 2, 3, 5, 8.
22. Herz. Ber., 31–2670; Seidell, Sol. of Chem. Subst.
23. Regnault, Landolt-Börnstein, 771.
24. Schiff, Landolt-Börnstein, 767.
25. Schiff, Phisico-Chemical Tab. (Castell-Evans), 197.
26. Battelli, Landolt-Börnstein, 770.
27. Wirtz, Smithsonian Phy. Tab., 214.
28. Schiff, Physico-Chem. Tab. (Castell-Evans), 417.

7. EFFECT ON GAS QUALITY

As has been stated previously, the removal of benzol and toluol from the gas reduces its heating value and candlepower. The amount of reduction will depend on the original quality of the gas before washing, the thoroughness of washing, and the process of gas making employed.

Operators of benzol-recovery plants differ considerably in their opinion as to just what the average reduction would be, but the general opinion seems to be that the complete removal of light oils from gas results in reduction of at least 50 per cent in the open-flame candlepower and from 1½ to 8 per cent in the heating value (averaging about 5 per cent). Various operators have endeavored to establish ratios between light-oil removal and heat reduction. One operator states that for every 0.1 of a gallon of light oil removed per 1000 cubic feet of gas, the heating value will be reduced between 13 and 14 Btu per cubic foot and the candlepower between 2½ and 3 candles. Another states that the reduction of heating value is 10 Btu for every 0.1 gallon of light oil removed.

If it is desired to recover toluol and still maintain a fairly high standard of gas quality, various methods may be employed, as follows:

1. The light oils may be entirely removed from the gas, fractionated, and the benzol fraction returned to the gas by some suitable method.

2. The gas may be only partially washed of its light oils by the use of insufficient wash oil for complete removal of the light oils.

3. All the light oils may be washed from the gas and no portion returned, the enrichment up to the desired quality being accomplished by the addition of volatile petroleum distillates.

The decision as to which method should be employed in a given case would depend upon several considerations. A partial scrubbing of the gas by process 2 would be most convenient for plants which did not fractionate the light oils, but the possible toluol recovery would be considerably less than in methods 1 and 3, since much of the toluol would necessarily remain in the gas. Method 3 would require the purchase of petroleum distillates, the price of which might make the method unprofitable. It is stated by some operators that more distillate must be added to gas than the quantity of light oil removed to compensate for its removal, especially where a candlepower standard is in force, since petroleum distillates do not contribute to the open-flame candlepower to anything like the extent that benzol does. In order to have the reenrichment effective the distillate would have to possess certain qualities. One operator states that it should volatilize completely below 150° C and have a heating value of at least 23 000 Btu per pound.

The gas quality standards in force in a given locality would also to a considerable extent determine the method of reenrichment and the toluol yields obtainable. A company which is operating under a very high candlepower standard is forced to reenrich heavily or to be satisfied with a small toluol recovery. A lower standard obviates this difficulty to a considerable extent. One operator in a large city which maintains a 22-candlepower standard states that a plant now making gas of that quality could, if relieved of the candlepower requirement, still maintain over 600 Btu with a recovery of 0.05 gallon toluol and 0.08 gallon benzol, even though no light oils were returned to the gas. In plants making other gas qualities, the yields of toluol and effect upon gas quality would, of course, be different.

Figures as to the value of the light oils in water gas from various plants indicate that for medium candlepower gas, viz, 12 to 17 candles, 0.1 gallon of benzol per 1000 cubic feet returned to the gas raises the candlepower about 2 candles. The effect on the heating value in these particular cases is not known, but it seems probable that this would give about 12.2 Btu per cubic foot as the increase in heating value due to the addition of 0.1 gallon of benzol per 1000 cubic feet of gas. This subject is discussed more fully in Part III of this paper.

B. CONSTRUCTION AND OPERATION OF LIGHT-OIL RECOVERY PLANT

In the following section it is impossible to describe in detail all types of recovery apparatus in use. An endeavor has been made to describe typical construction, but dimensions and details where given should be understood to apply only to certain installations observed.

1. SCRUBBERS

The apparatus in which the gas is brought into contact with the washing oil is known as the "scrubber" or benzol washer. In different plants it assumes different forms; that is, it may be of the rotary type, some form of bubble type, the spray type, or the tower and hurdle type.

(*a*) *Rotary Scrubbers.*—A rotary scrubber may be constructed somewhat as follows: The shell is of cast iron approximately cylindrical in shape, with the axis parallel to the floor. It is divided into a number of compartments by means of plates, which are the full size of the cross section of the shell and which have circular openings at their centers. A second set of plates placed alternately with the first reach from the bottom up to the middle of the shell.

The shell is traversed lengthwise by a shaft, the axis of which coincides with the axis of the shell, and which is supported by suitable bearings on the end plates and on the second set of cross plates mentioned above. On the shaft are fastened gas baffles in the form of disks made up of wooden slats, there being one disk for each compartment of the shell built up so that the gas can travel between the slats either from circumference of the shell to the center or vice versa, but not straight across the disks parallel to the shaft. The central openings of the disks are alternately closed and opened in such a manner in conjunction with the arrangement of the cross plates that, when the scrubber is filled with the washing medium to the proper level, the gas is forced to travel through the upper half of the disks from circumference to center and from center to circumference alternately. A small engine, or some other motive power, is used to rotate the shaft and the attached disks.

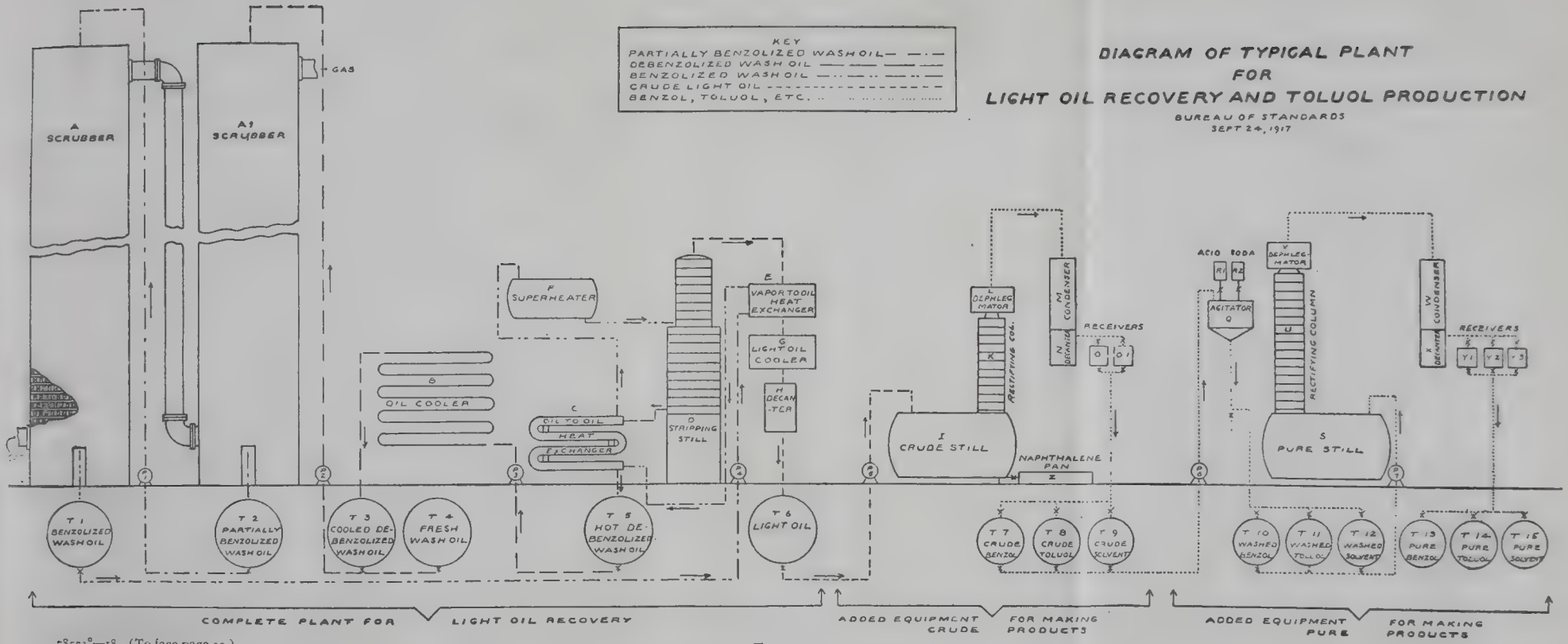
The spaces between the slats are very narrow, and thus the gas in passing through the scrubber flows in thin streams over surfaces which are kept continually wet, as the shaft rotates by dipping into the washing medium in the lower part of the shell. The

washing medium is admitted to the end opposite that at which the gas enters, and is carried by suitable overflows from one compartment to the other until it finally leaves the scrubber at the gas-inlet end. In this way the gas most thoroughly washed comes into contact with the freshest oil.

(*b*) *Bubble-Type Scrubbers.*—A scrubber of the bubble type usually consists of a series of superimposed sections constructed of cast iron. Each section has one or more circular openings raised above its bottom and covered with hoods or bells, the edges of which are usually serrated or slotted so that any gas flowing through the opening can only pass out under the edge of the hood, which edge is kept sealed with the washing medium. The serrations or slots break up the gas stream into small bubbles, giving thorough contact with the washing medium. Likewise an overflow from each section to the section below is provided. The gas enters the bottom of the washer and, passing up, bubbles through the seal of wash oil in each section. Fresh wash oil is introduced at the top and fills the top tray to the height of the overflow pipe, through which it drains to the next section below, and so on down through the sections. The process is therefore so arranged that the gas most nearly washed is brought in contact with the freshest oil.

(*c*) *Spray-Type Scrubbers.*—In the spray type of scrubber the wash oil enters the top section of a series of superimposed sections through a siphon and passes through a spraying cone, by which it is hurled by centrifugal force out through the perforations in the cone in a very fine mist or spray, thus filling the entire gas space with a mist of wash oil. The oil flows into the next lower section through the gas ports and is again brought into contact with the gas in the same manner here, finally leaving the bottom of the washer in a saturated condition. The gas enters from the bottom and passes upward successively through the washing sections. The power supplied to the centrifugal spraying cones is usually furnished by a small steam engine.

(*d*) *Hurdle-Tower Scrubbers.*—The hurdle-tower scrubbers in use at the present time usually consist of cylindrical steel shells about six to seven times as high as the diameter. Two such shells are shown as *A* and *A 1*, Fig. 1. These shells contain a great number of grids or trays, commonly called "hurdles," made of white-pine slats 5 or 6 inches high by 1 inch wi le. Except for spaces at the bottom and top of the scrubber these grids are practically superimposed one upon the other, with the openings staggered, separated by small spacing strips.



78573°—18 (To face page 22.)

FIG. 1

At the top of the tower the wash oil is sprayed in from a number of nozzles. After passing through the interstices in the grids it collects at the bottom in a reservoir. The gas enters the tower at the bottom and rises through the interstices in the grids, by this means coming into contact with the washing medium. Thus the freshest wash oil introduced at the top comes into contact with the most nearly washed gas.

In one plant designed to scrub about 10 000 000 cubic feet of gas per day in which the tower is about 75 feet high and 11 feet in diameter, there are 84 grids, arranged in 4 banks of 21 each. The space at the bottom of the tower is about 8 feet high; that at the top is about 5 feet. Spaces of about 3 feet separate the various sets of trays. In the top are 12 oil nozzles. The foundation of such a tower is constructed of concrete and extends down about 7 feet. The size of the washer is, of course, dependent upon the capacity of the plant and the design of the washer. In hurdle-type scrubbers 30 cubic feet of scrubber capacity per 1000 cubic feet of gas washed per hour is a common figure.

In some plants, notably in those utilizing existing tanks or towers, the grids are replaced by trays on which coke is piled or by one tray set near the bottom of the tower on which coke is piled to nearly fill the tank.

The wash oil is pumped by a circulating oil pump from the oil-storage tank T^4, Fig. 1, or circulating tank T^3, Fig. 1, to the nozzles in the top of the scrubber. From these nozzles the oil is sprayed into the scrubber at a temperature of 30° C or lower. When two or more scrubbers are placed in series, the wash oil which has passed through the last scrubber is usually pumped to the top of the next preceding scrubber and passes down through this in the manner previously described. The gas passes from the top of the first scrubber to the bottom of the second and up through the second scrubber. An additional oil pump is required for each tower scrubber which is being used.

2. HEAT EXCHANGERS

In order to utilize the heat in the vapors and wash oil leaving the stripping still, which would ordinarily be wasted, several pieces of apparatus, known as heat exchangers or interchangers, may be used. One of these is known as the "vapor-to-oil" and the other as the "oil-to-oil" heat exchanger. Each of these may assume a different form in different plants, or the order of succession may be different. In some plants one or the other may

be eliminated, and in very small plants, especially those which are homemade, both are frequently missing.

(*a*) *Vapor-to-Oil Heat Exchanger.*—The wash oil containing the light oils is pumped from the bottom tank of the tower scrubber to the vapor-to-oil heat exchanger, shown as *E*, Fig. 1. This piece of apparatus usually consists of a cylindrical steel shell which contains a number of tubes. In one plant designed to scrub 10 000 000 cubic feet per day it is about 8 feet long and 4 feet in diameter, containing about 150 tubes 2 inches in diameter. The cold benzolized wash oil flows through the tubes, while hot vapors from the top of the continuous still (hereafter described) pass around the tubes, being directed by a series of baffle plates. At the outlet of this heat exchanger the temperature of the benzolized wash oil is about 72° C. A heavy covering of heat insulating material aids in the conservation of the heat.

(*b*) *Oil-to-Oil Heat Exchanger.*—After leaving the vapor-to-oil heat exchanger the benzolized wash oil passes to and through an oil-to-oil heat exchanger, shown as *C*, Fig. 1. In some plants this apparatus is an oblong box, in which the hot debenzolized wash oil from the still passes through pipe coils, the benzolized oil passing around the outside of these coils. In other plants this apparatus is built up of a number of pipe sections, joined at the ends. Each pipe contains a number of smaller pipes. The hot debenzolized oil from the base of the still passes through the smaller pipes, the benzolized oil passing around them. The larger pipes are arranged in several banks one above the other. The banks are connected together at each level, but the superimposed pipes are only connected in pairs.

In one plant of the size previously mentioned this heat exchanger is constructed of eight 10-inch pipes heavily covered with insulating material. Each of the large pipes contains 14 one-half-inch tubes. The over-all length is about 26 feet, the height about 7 feet, and the width about 3 feet.

At the outlet of this heat exchanger the temperature of the benzolized wash oil is about 94° C and that of the debenzolized wash oil about 87° C.

3. SUPERHEATER

Passing from the oil-to-oil heat exchanger the benzolized wash oil enters a superheater, sometimes called a preheater, shown as *F*, Fig. 1. This piece of apparatus usually consists of a cylindrical tank made of steel. Inside of the shell are a number of

small tubes and several baffle plates, the benzolized wash oil flowing around and steam passing through the tubes. In the plant previously mentioned this superheater is about 10 feet long and 4 feet in inside diameter. In this shell are about one hundred and sixty 2-inch tubes. The superheater is heavily insulated, and, owing to great corrosive action, a duplicate is usually provided. Likewise the parts are so arranged as to be removable without great difficulty. A safety valve is placed on the top of this piece of apparatus.

The temperature of the benzolized wash oil leaving the superheater is usually about 145° C, although this temperature varies in different plants.

4. CONTINUOUS STRIPPING STILLS

After the benzolized wash oil leaves the superheater, it passes into the continuous stripping still, in which the wash oil is freed from practically all of the entrained light oils. This still, shown as *D*, Fig. 1, is usually constructed in two main portions, each of which is built up of superimposed individual sections. In some types the total height is about four to five times the diameter of the lower portion, while the diameter of the upper portion is usually about three-fourths that of the lower. These proportions, however, may vary considerably. Each section is made of cast iron and is from 12 to 14 inches in height. The upper portion of the still usually has about half as many sections as the lower and acts as a partial rectifying column assisting in retaining some of the wash oil in the still, which might go over with the light-oil vapor.

In construction these sections are similar to those described under the bubble type of scrubbers, there being a number of openings arranged in a circular manner in each section.

The benzolized wash oil enters the still at the base of the upper portion and passes down through the large portion of the still, rapidly giving up the light oil which it contains. Steam is admitted at the bottom and passes up through the still, bubbling from under the sealing bells of each tray and carrying upward the light oil in the form of vapor from the wash oil which seals the bells. The steam and these vapors pass through each individual section of the still in the manner just described and mingle in the upper sections of the still with the vapors set free there.

In one city gas plant designed for 10 000 000 cubic feet per day the still is about 27 feet high. The lower portion is about 6 feet in diameter and the upper about 4½ feet in diameter. The lower

portion consists of about 12 sections and the upper of about 6 sections.

The temperature of the light-oil vapor leaving the top of this still is about 104° C, while the wash oil, stripped of all light oils, leaves the bottom at a temperature of about 130° C. In some types of light-oil recovery plants the light-oil vapors after leaving the top of the still column pass through some type of dephlegmator, through which a regulated amount of cooling water in separate compartments is flowing, so that while the light oils are permitted to pass uncondensed the water vapors and any wash oil vapors present condense out and run into a collecting tank. From this tank the water is drawn off and the wash oil, which contains some light oil, is pumped again with the benzolized oil into the still, or, if too heavy to be handled by the stripping still, it is sometimes put into the light oil to be subsequently refined in the crude still. Some operators claim that by using steam sufficiently superheated in the stripping still the condensation of steam in the wash oil may be largely avoided, nearly all the steam going forward and condensing with the light oils, from which it is more readily separated.

5. WASH-OIL COOLER

The debenzolized wash oil leaving the base of the continuous still passes into the oil-to-oil heat exchanger, where its temperature is lowered to about 87° C. From this piece of apparatus the wash oil drains to a hot-oil drain tank T_5, Fig. 1, after which it is pumped through a wash-oil cooler, shown as B, Fig. 1. This consists of a number of pipe coils, the cooling water being showered upon the outside of the coils, while the wash oil flows through the inside. Another form of cooler permits the water to flow through the pipes, the oil flowing on the outside.

In the plant previously mentioned these coils are made up of 2-inch pipe with return bends at the ends, the whole being about 10 feet high. At this plant there are six of the sets of coolers parallel to each other.

In some types of recovery plants the wash-oil cooler consists of pipe coils immersed in a tank of water. This apparatus is sometimes designated as the temperature regulator, since it determines the approximate temperature at which the oil is to enter the gas scrubbers.

From these coolers the wash oil passes to a wash-oil storage tank shown as T_3, Fig. 1, from which it is pumped to the top of the tower scrubber to again pass through the system.

In some plants the wash oil may be pumped directly from the oil-to-oil heat exchanger through the coolers and into the wash-oil circulating tank.

6. CONDENSER AND SEPARATOR

The crude light-oil vapors leaving the top of the continuous still with a temperature of about 104° C pass into the vapor-to-oil heat exchanger before described. From here they pass into a condenser, shown as *G*, Fig. 1, which is usually of cylindrical shape. Inside of the outer shell are a number of tubes around which the vapors pass, the water passing through the tubes. If this piece of apparatus consists of a worm type of condenser, the vapors pass through the coil which is surrounded by the cooling water. The light oils leaving the condenser have a temperature of about 30° C. In the plant previously mentioned this condenser is about 9 feet long and 4 feet in diameter.

In many large plants it is found advantageous to install a pipe connection with a small blower or steam jet between the outlet of the light-oil condenser and the outlet of the last gas scrubber. By this means a considerable amount of noncondensible light vapors leaving the stripping still may be returned to the gas, thereby reenriching it to a considerable extent.

From the light-oil condenser the light oil enters a separator, or decanter, shown as *H*, Fig. 1. Here any water entrained in the light oil is decanted or separated due to the difference in specific gravity. This piece of apparatus usually consists of a small cylindrical shell provided with two outlets, one for oil and one for water. The water connection extends down inside of the shell nearly to the bottom, the oil outlet being close to the top. The water passes off to a sewer and the crude light oils pass either to a crude light-oil storage tank, shown as *T6*, Fig. 1, or directly into the crude rectifying or "boiler" still, if the plant is so arranged as do away with this tank.

7. CRUDE RECTIFYING STILLS

From the crude light-oil storage tanks, the crude light oil is pumped into the crude rectifying still or boiler still. This still is composed of three parts, the boiler, shown as *I*, Fig. 1, the rectifying column *K*, and the dephlegmator, *L*.

The boiler still consists of a cylindrical shell having bumped heads, internally braced, or otherwise strengthened against collapse. It is designed to handle from 5000 to 12 000 gallons or

more of crude light oil, according to the size of the plant. The shell is covered with insulating material in order to reduce heat losses. The distillation is carried out by the use of pressure steam in coils placed in the bottom of the still. There is also a perforated pipe in the bottom of the boiler so arranged that live steam can be utilized to aid in the distillation, if desired, although this latter is not frequently made use of. Vacuum connections are also provided so that the distillation may be aided in the latter part of the cycle.

In one plant designed to wash 10 000 000 cubic feet of gas per day this still, designed to handle a charge of 5000 gallons of crude light oil, is made of steel, 7 feet 6 inches in diameter and 16 feet long over-all. The steam coils are made of 2-inch pipe, and a ball-and-lever safety valve is attached to the shell.

The crude light oils are pumped into the boiler still until its working capacity is reached, the distillation being carried out at the temperature previously described. (See p. 16.) The residue which remains in the still consists of wash oil, naphthalene, etc., and is drained to the naphthalene pans, which are described later.

After the charge is fractionated a fresh charge is placed in the still. The time required to complete a run varies. In some plants 40 hours are required while in others 24 hours are deemed sufficient.

The vapors from the still pass into the rectifying column, shown as *K*, Fig. 1, as they are distilled off. This column consists of a number of cast-iron sections, mounted one upon the other, each section being from 12 to 14 inches in height. It is stated by some operators that the sections must be at least 10 inches in height to prevent vapors passing over as a spray. The column is mounted upon the top of the boiler still close to one end in some types of apparatus, while in others it stands on a separate foundation. The column is usually about three times as high as it is in diameter, being heavily lagged with insulating material. In construction each section is similar to those already described. There are a number of "up" openings in each section, the covering bells having their edges serrated in some cases, while in other cases the edges of the bells are perforated or slotted. The vapors rise through the openings and bubble through a seal composed of the descending oils which are condensed in the dephlegmator and which drain back down the rectifying column, becoming heavier as they near the bottom of the column.

In one plant, washing about 10 000 000 cubic feet of gas per day, this rectifying column is about 12 feet 6 inches high and 4 feet in diameter and is built up of 10 sections.

Mounted on top of the rectifying column or on a separate foundation is the dephlegmator (called the "planer" by some operators), shown as *L*, Fig. 1. Here the vapors rising from the rectifying column are cooled by a circulation of water. The heavier portions condense and drain back down the rectifying column as before described.

The dephlegmator usually consists of a cylindrical or box-shaped shell containing a number of water tubes. Baffles direct the course of the vapors through the dephlegmator. In the particular plant above mentioned the dephlegmator is about 5 feet 6 inches long and 3 feet 6 inches high and wide, and it contains about 148 water tubes, each 1¾ inches in diameter.

In another plant one rectifying column and dephlegmator is provided for two boiler stills.

In some cases the crude rectifying still and stripping still may be combined. In plants producing from 15 to 40 gallons of light oil per hour (about 1 000 000 to 3 000 000 cubic feet of gas per day) it may be found advantageous to have the stripping still of such size that it can strip in, say, 16 hours all the benzolized wash oil produced in 24 hours, and to have such accessories to the still provided that it can be used as a crude rectifying still during the remaining eight hours of the day. When a still is to serve both purposes, it should usually be equipped with some form of dephlegmator and with an auxiliary still base which can be valved off and used as a light-oil storage tank while the still column is stripping wash oil. This still base is equipped with closed steam coils and a live-steam connection, and is so connected with the still column that when in use rectifying light oil it corresponds in effect to the boiler part of a regular rectifying still. In plants producing more than 40 gallons of light oil per hour it would probably be more practicable to have entirely separate rectifying and stripping stills. In plants producing less, and probably in some producing more than 15 gallons of light oil per hour when advantageously situated, it would usually be impracticable to attempt fractionation of the light oil at all at the gas works. A diagram showing the arrangement of such a plant is reproduced for the Report of the Subcommittee on Coal Tar By-Products, published September, 1917, shown as Fig. 2.

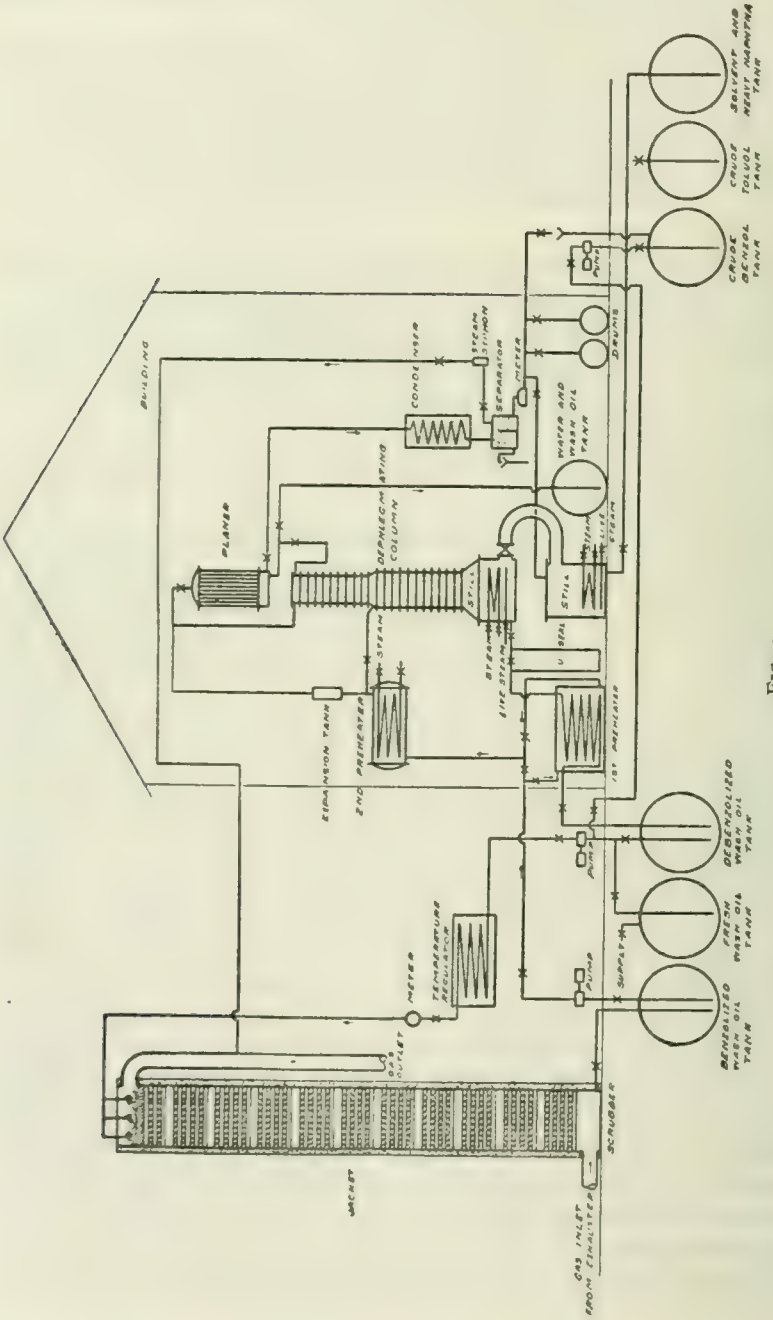

BUILDING
CONDENSER
STEAM SILENCER
SEPARATOR
WATER AND WASH OIL TANK
DRUMS
PUMP
CRUDE BENZOL TANK
CRUDE TOLUOL TANK
SOLVENT AND HEAVY NAPHTHA TANK
PLATES
DEPHLEGMATING COLUMN
STILL
STEAM
LIVE STEAM
COIL
EXPANSION TANK
ENG. PREHEATER
1ST PRECIPITATOR
DEBENZOLIZED WASH OIL TANK
FRESH WASH OIL TANK
PUMPS
SUPPLY
HEATER
TEMPERATURE REGULATOR
GAS OUTLET
BENZOLIZED WASH OIL TANK
SCRUBBER
GAS INLET
FROM CONDENSER
WASH

FIG. 2

8. NAPHTHALENE PANS

The still residue, containing wash oil, naphthalene, etc., is drained from the bottom of the boiler still into the naphthalene pans, shown as *Z*, Fig. 1. Here the naphthalene separates out on cooling in the form of crystals and the wash oil drains back to the circulating wash-oil tank. The naphthalene may be further dried by using a centrifugal machine.

In plants scrubbing the light oils from water gas little naphthalene will usually be encountered.

9. CONDENSER AND SEPARATOR

The benzol, toluol, and solvent naphtha vapors leaving the dephlegmator enter a condenser, shown as *M*, Fig. 1. This is a cylindrical tank inside of which are a number of tubes through which water circulates. A series of baffle plates direct the flow of vapor back and forth at right angles to the tubes. In this condenser the various light-oil vapors are condensed into liquid form.

In one plant handling 10 000 000 cubic feet of gas per day this apparatus is about 8 feet 6 inches high and 3 feet in diameter, and contains about 90 tubes, each 1½ inches in diameter.

From the condenser the benzol, toluol, etc., pass to a water separator, shown as *N*, Fig. 1, which is similar to the one described previously.

10. RECEIVERS OR SAMPLING TANKS

From the separator the benzol, toluol, etc., drains into one of two receivers, shown as *O* and *O1*, Fig. 1. These are small graduated cylinders of about 100 gallons capacity and are used as sampling tanks. One may be filling while a sample from the other is being tested to determine its character.

From these receivers the crude benzol, toluol, and solvent naphtha is drained into the proper one of the crude storage tanks, shown as *T7*, *T8*, and *T9*, Fig. 1.

11. AGITATOR

If the plant is one which produces chemically pure products, the crude benzol, toluol, or solvent naphtha is pumped from the crude storage tanks into an agitator or chemical washer, shown as *Q*, Fig. 1. This agitator is a large lead-lined, vertical, cylindrical, steel vessel with a conical bottom, and contains power-driven paddles. In one plant the agitator is about 8 feet in diameter and 6 feet high.

In the agitator the benzol or toluol, etc., is washed with sulphuric acid, which is supplied from a small storage tank shown as

R, Fig. 1. The paddles are located at a height which permits of discharging the acid in the bottom of the washer near the level of the benzol or toluol, and the distribution of it in such a manner as to obtain a thorough mixture. This mixing or washing is carried out for 30 to 90 minutes, after which the agitator is shut down and about 30 minutes are allowed for the used acid to settle to the bottom of the agitator. The acid is used to separate the unsaturated hydrocarbons, principally olefines, which settle with the residue to the bottom of the agitator. The sludge is run off and the acid contained in it is regenerated for other uses. The benzol or toluol is washed with water, after which a caustic-soda solution is run into the agitator to neutralize any remaining traces of the acid in the benzol or toluol. This latter solution is stored in the tank shown as *R1*, Fig. 1. After properly mixing with the light oil the soda is allowed to settle and is then drained off, the benzol, or toluol being again washed with water to remove any traces of caustic soda. From the washer the washed benzol, toluol, or solvent naphtha drains into the washed-oil tanks, shown as *T10*, *T11*, and *T12*, Fig. 1. If too much acid is used at one time or the time of stirring is too long the sludge will be lumpy.

12. RECTIFYING STILL

From these tanks the washed benzol, toluol, or solvent naphtha is pumped into the pure rectifying still. This still consists of three parts—the boiler still, shown as *S*, Fig. 1, the rectifying column, *U*, and the dephlegmator, *V*. Each of these three parts fulfills the same function, and is similar in construction to those described for the crude rectifying still. However, the rectifying column is usually considerably higher than that on the crude still, in order that closer fractionation can be accomplished. If benzol is introduced into the still pure benzol is distilled over at a temperature between 80 and 81.5° C. If toluol is introduced into the still, pure toluol will distill over between 110 and 111.5° C. However, these temperature ranges may be varied to suit the requirements of the users of the pure products, but the true boiling points (benzol 80.4° C, toluol 110.3° C) should always be included within the range chosen. The time of distillation is usually about five times as long as in the crude still, and the size of the still is usually greater.

The residue is usually returned to the tanks containing the washed products, while the vapors leaving the dephlegmator pass through a condenser, shown as *W*, Fig. 1, and then through a water separator, shown as *X*. These two pieces of apparatus are similar in all respects to those described previously.

From the separator the C. P. benzol, C. P. toluol, or solvent naphtha is run into one of three sampling tanks, shown as *Y*, *Y1*, and *Y2*, Fig. 1, similar to those marked *O* and *O1*, after which it passes to the pure tanks, shown as *T13*, *T14*, and *T15*, Fig. 1.

13. RECOMMENDATION OF COMMITTEE ON CHEMICALS

The following tabulation is reproduced from the Report of the Subcommittee on Coal-Tar By-Products for the committee on chemicals, published in September, 1917, presenting in complete form the apparatus required, together with sizes, for plants of various capacities. This report is given in detail in the Gas Institute News, November, 1917.

TABLE 3.—Detail Sheet of Light-Oil Recovery and Fractionating Plants

	No. 1, 150 gallons light oil per hour	No. 2, 100 gallons light oil per hour	No. 3, 65 gallons light oil per hour	No. 4, 40 gallons light oil per hour	No. 5, 25 gallons light oil per hour	No. 6, 15 gallons light oil per hour
Oil temperature regulator heating surface (square feet)	1650	1100	687	412	247	165
Benzolized wash-oil tank (gallons)	10 000	8000	6000	10 000	3000	2000
Oil-to-oil heat exchanger heating surface (square feet)	900	600	375	300	135	90
Steam preheater for oil-heating surface (square feet)	1800	1200	750	600	270	180
Debenzolizing columns, number of top sections	8–28″	6–28″	6–28″	20–28″	12–28″	9–28″
Debenzolizing columns, number of bottom sections	18–42″	14–42″	16–28″	None	None	None
Planer debenzolizing column cooling surface (square feet)	600	400	250	200	90	60
Condenser cooling surface (square feet)	750	500	300	250	112	75
Water separator (inches)	24x48x36	24x36x36	24x27x27	20″ pipe	10″ pipe	10″ pipe
Light-oil storage tank (gallons)	10 000	7200	4500	1000	4000	4000
Debenzolized wash oil tank (gallons)	10 000	8000	6000	10 000	3000	2000
Storage tank for reserve straw oil (gallons)	10 000	8500	6500	.....	.....	.....
Cooling pans for removing resin (gallons)	1000	700	500	500	300	200
Capacity of fractionating still body (gallons)	6000	4000	2500	[a] 1200	.....	.....
Heating coils, heating surface (square feet)	600	400	250	250	.....	.....
Column	22–42″	12–42″ / 10–28″	22–28″	None	None	None
Planer cooling surface (square feet)	375	250	156	.....	.....	.....
Condenser cooling surface (square feet)	375	250	156	.....	.....	.....
Water separator	16″ pipe	12″ pipe	10″ pipe	8″ pipe	None	None
Crude benzol tank (gallons)	10 000	8500	6500	6500	.....	.....
Crude toluol tank (gallons)	10 000	8500	6500	6500	.....	.....
Residue tank (gallons)	10 000	8500	6500	6500	.....	.....
Pumps (capacity, gallons per minute):						
Oil to primary scrubber	450	300	195	40	25	15
Oil to secondary scrubber	150	100	65	.....	.....	.....
Oil to debenzolizing still	150	100	65	40	25	15
Light oil to still	75	50	32	.....	.....	.....
Reserve	150	100	65	40	25	15

[a] Attached to debenzolizing column.

TABLE 3.—Detail Sheet of Light-Oil Recovery and Fractionating Plants Contd.

	No. 1, 150 gallons light oil per hour	No. 2, 100 gallons light oil per hour	No. 3, 65 gallons light oil per hour	No. 4, 40 gallons light oil per hour	No. 5, 25 gallons light oil per hour	No. 6, 15 gallons light oil per hour
Thermostats: Preheater 240°	1	1	1	1	1	1
Planer 210°	1	1	1	1	.....	.....
Temperature regulator 120°	1	1	1	1	1	1
Meters (gallons per minute):						
Scrubbing oil, disk type	85	55	40	30	15	10
Light oil, condensation type	200	150	100	60	40	25
Distillate oil, indicating type	400	300	200	100	.....	.....
Thermometers: Recording	7	7	7	5	.....	.....
Indicating	12	12	12	8	8	8
Pressure gauges: Indicating	14	14	14	14	10	10
Piping: Scrubbing oil (inches)	4-7-10	2½-5-8	2-4-6	1½-3-4	1½-2½-4	1-2-3
Vapor (inches)	6	4	4	3	3	3
Light oil (inches)	2½	2	1½	1½	1	1
Steam supply (inches)	4	3	2½	2	1½	1½
Water supply (inches)	3	2½	2	2	1½	1½
Steam required (max. pounds per hour)	8250	5500	3580	2200	1370	825
Water required (max. gallons per hour)	5250	3500	2280	1400	875	525

14. LOCATION RELATIVE TO EXISTING PLANT

The relative position occupied by the light-oil recovery plant in reference to the remainder of the manufacturing plant varies in different localities. In some plants the washing of the gas takes place before the gas goes to the purifiers, while in others after the gas passes the purifiers. However, in any plant in which quantities of tar and ammonia are produced, the oil washing of the gas should take place after the tar and ammonia are removed.

The relative arrangement of the equipment depends largely upon the existing manufacturing plant. If possible, it should be close to the necessary steam and water supplies, and all of the parts should be so arranged that there would be only the minimum of all classes of piping required.

15. SPACE REQUIRED

Sufficient space is required for the necessary number of tower scrubbers, if the plant is to be of this type, for the building to house the stills, pumps, and other necessary accessories, and for the storage and circulating tanks and other outdoor equipment. The entire recovery plant should be in as compact form as possible, yet should not be too crowded.

The amount of floor space which is required for the building in which the stills are located and in which some of the accessories must be placed depends largely upon the products recovered and the daily capacity of the plant. Sufficient floor space must be provided for the stills, and the pumps should also be provided for in estimating the amount of floor space required. It is possible to put much of the remaining apparatus at a higher level, thus economizing in floor space.

It is impossible to state with any degree of exactness the floor space requirements, but in several plants from 2400 to 3000 square feet of floor area are required for the recovery of C. P. products. In another plant 1200 square feet are required for the recovery of crude benzol, toluol, etc.

16. STORAGE OF MATERIALS

The question of storage of materials, wash oil, both benzolized and debenzolized, the crude light oil produced in the continuous stripping still, the crude benzol, toluol, solvent naphthas, etc., the refined products if the plant carries the process to this extent, is one which must be considered carefully. Lack of adequate means of storage often means the discontinuance of the plant, unless it is possible to utilize some other equipment in the plant.

In all plants an extra supply of wash oil must be stored. It is advisable to have as a reserve from one-half to the full amount of wash oil circulated through the system in one day. This preferably should be stored in one tank, which should have a capacity at least equal to twice that of a tank car, so that the unloading of incoming cars may be done promptly. It is also advisable to have sufficient tank capacity for all the wash oil being utilized in the system, one-half for the benzolized wash oil and one-half for the debenzolized wash oil.

It is also necessary to provide a tank for the light oil recovered from the continuous still. This tank should have a capacity equal to that of the fractionating still unless there are two fractionating stills with one rectifying column, as is the case in some plants. If the plant is not equipped for fractionating the light oil, sufficient storage capacity to tide over any interruption of shipping facilities should be provided. It is suggested that capacity for a week's production should be available, but where practicable the capacity should be not less than twice that of an average tank car.

If the plant recovers crude benzol, toluol, etc., but does not further fractionate these, sufficient storage capacity should be provided to care for each of the crude fractions, so that no danger of a lack of storage will be met. In several plants the storage capacity for benzol is about twice that of the toluol, solvents, etc. It is suggested that capacity for a week's output of each of these liquids be provided. If the plant produces C. P. products, sufficient storage capacity for each of the crude liquids should be provided, so that the pure still will be able to operate long enough to refine the preceding crude fractions.

The C. P. benzol, toluol, etc., likewise requires sufficient storage space, so that if any interference with shipments occurs it will not be necessary to curtail the operation of the plant in any way. Here also the benzol storage should be about twice that for the toluol. In addition, in a C. P. plant storage must be provided for the sulphuric acid. As a summary, approximately the storage capacity indicated is required for the following materials:

When crude light oil only is recovered—

Old wash oil debenzolized.

(Equal to one-half the amount in circulation.)

Old wash oil benzolized.

(Equal to one-half the amount in circulation.)

New wash oil.

(From one-half to full amount in circulation; at least two carloads.)

Crude light oil.

(Equal to capacity of crude still. If no fractionation, should be large enough to avoid difficulties caused by shipping delays; at least two carloads.)

When crude benzol, toluol, etc., are recovered—

Same as above, and crude toluol, crude benzol, crude solvent naphthas, heavy naphtha, and crude intermediates.

(Benzol storage about twice as great as other fractions. If further refining is done, should be large enough to allow refining stills to work necessary time. If no refining, tanks should be large enough to avoid difficulties caused by shipping delays.)

When C. P. products are recovered—

Same as previously stated, and washed benzol, washed toluol, and washed solvent naphthas.

(Benzol about twice as great as other fractions. Sufficient capacity to allow refining still to operate requisite length of time.)

Sulphuric acid, pure toluol, pure benzol, and pure solvents.

(Benzol about twice as great as other fractions. Large enough to avoid difficulties caused by shipping delays.)

In addition, one or two spare tanks would be advisable.

The location of these tanks is largely dependent upon local conditions. In some plants they are placed underground; in other plants they are located in water-tight pits, while in still other plants part or all of the tanks are above the ground surface. They

may be grouped together or they may be separated into different groups, largely depending on the amount of available space or local fire regulations.

17. SAFETY IN OPERATION

The buildings for a light-oil recovery plant should be located as far as practicable from other plant structures; or, if sufficient distance is not obtainable owing to existing conditions, the building should be made an individual unit by bricking up or otherwise closing all openings into adjacent buildings. Under any conditions, it is desirable that the stills be located about 200 feet from any source of flame, since the heavy benzol vapor is quite prone to travel along the ground for a considerable distance without dilution to a sufficient degree to prevent inflammability.

The building should be constructed of fireproof material throughout, and should be well ventilated and equipped with wire glass windows and metal fire doors. If practicable, it should be located away from railroad tracks or sidings, and the loading platform should be removed as far as practicable from the rest of the plant.

No open flames should be brought near any portion of a light-oil recovery plant, and only incandescent electric lamps, incased in vapor-proof globes with a protective wire casing around the globe, should be used around any portion of a light-oil plant. In case portable lights are required, small storage battery lights should be used. All electric wiring throughout the plant should be incased in iron-pipe conduits, and no extension cords should be used.

A sufficient number of fire extinguishers of the type capable of extinguishing an oil fire and buckets containing clean dry sand should be conveniently placed, not only inside of the building, but also outside of the building. A plentiful supply of water, under high pressure, should also be readily accessible, and an efficient corps of men trained in fire extinguishing should be available at all times.

Smoking should not be permitted in or near the light-oil plant at any time.

All stills and superheaters should be equipped with a positive safety-valve set to blow at about 10 pounds pressure. Likewise all tank cars and tank wagons should be equipped with safety valves. All tanks should vent to the atmosphere, the vents being provided with a screen of fine wire in order to prevent the ignition of the vapors by sparks.

All joints in the piping, stills, tanks, or any other apparatus should be kept tight, so that leaking vapors do not accumulate in the buildings or their vicinity. All condensers should be supplied with adequate cooling water to prevent the escape of vapor into the air. Inclosed tail boxes, freely vented outside of the buildings, should be used.

When necessary to clean stills special precautions are necessary to prevent the workmen from being asphyxiated. No one should enter the stills until they have been thoroughly purged of all vapors and are cool. All connecting valves should be closed and locked, and if the valves are not tight, the piping should be disconnected. At least two openings should be maintained in the still to allow a circulation of air throughout. The man entering should be provided with a life belt and rope, and another man should be stationed outside of the still to assist in case of necessity. He should obtain additional assistance before entering the still to aid the man overcome by the vapors in the still.

18. TECHNICAL CONTROL

In the operation of a light-oil recovery plant, in order to obtain efficient results and good quality of product, an adequate laboratory equipment must be maintained and at least one observer skilled in making the necessary tests must be employed. The extent of the tests in a given plant will depend upon the final products produced by the plant. Obviously a plant producing pure benzol, toluol, etc., will require much more elaborate tests than a plant selling unrefined light oil only.

The following is a list of tests usually required in the operation of a pure-products plant as furnished by the chief chemist of a large operating company:

A. Tests of Gas.

1. Determination of heating value and candlepower of gas entering the scrubbers.
2. Determination of light oil in gas entering scrubbers.
3. Determination of heating value and candlepower of gas leaving the scrubbers.
4. Determination of light oil in gas leaving scrubbers.

B. Tests for Wash Oil Still Operation.

1. Determination of light oil in benzolized wash oil.
2. Determination of light oil in debenzolized wash oil.
3. Tests of light oil.
   (*a*) Boiling point.
   (*b*) Determination of wash oil.

C. Tests for Crude Still Operation.

1. Receiver tests, i. e., boiling-point tests made to control fractionation.
2. Boiling-point tests of fractions (crude benzol, crude toluol, etc.) and residues sampled from their respective storage tanks.

D. Tests for Agitator Operation.
1. Distillation and acid tests of washed benzols.
2. Tests for SO_2 in washed benzols.
3. Specific gravity of regenerated sulphuric acid.

E. Tests for Pure Still Operation.
1. Receiver tests, i. e., boiling-point tests made to control fractionation.
   (*a*) Boiling point.
   (*b*) Acid test.
2. Tests of pure products sampled from storage or running tanks or from shipments.
   (*a*) Boiling point.
   (*b*) Acid test.
   (*c*) Specific gravity.
   (*d*) Freezing point (occasional in case of benzene).
3. Boiling-point tests of still residues.

F. Tests of Materials Used in Operation.
1. Wash oil.
   (*a*) Specific gravity.
   (*b*) Viscosity.
   (*c*) Emulsification.
   (*d*) Cold test.
   (*e*) Distillation.
   (*f*) Olefines.
2. Sulphuric acid, specific gravity.
3. Soda, Na_2O.

It is evident that in a plant shipping light oil away for further refining many of these tests could be omitted. In a very small plant, after the routine was established, occasional tests on gas before and after scrubbing and of the wash oil and light oil would probably suffice.

19. ADAPTATION OF LIGHT-OIL RECOVERY TO SMALL PLANTS

While the somewhat complicated system of heat exchangers and the careful design of stills, described in connection with the layout of a typical light-oil recovery plant, are very important in effecting economy of operation, they are not absolutely essential to the recovery of this material. The essential equipment of a small plant recovering only light oils consists of some form of scrubber to oil-wash the gas, a still for stripping the wash oil, a condenser for condensing the light oils, and some tanks for the storage of the various materials. A plant which is so fortunate as to possess or to be able to obtain cheaply some old gas-making or power-plant equipment might find it profitable to construct and operate a small plant. An old ammonia washer, water-gas scrubber, shavings scrubber, or even an old water-gas generating shell could be readily adapted for use as an oil washer by filling with wood grids, coke, or other materials which would furnish a large surface wet with the wash oil for contact with the gas. For

a stripping still an old boiler surmounted by a section of large-diameter pipe filled with stones could be used and the process made an intermittent one. In this case tanks would, of course, be necessary so that a portion of the wash oil could be circulated while another portion was being debenzolized and cooled. An old feed-water heater of the type in which the exhaust steam comes into direct contact with the cold water might be used as a continuous still by connecting the top with a condenser of some kind and admitting the wash oil near the top and live steam near the bottom. For a condenser a coil of pipe in a tank of water, an old closed coil-feed water heater, or similar apparatus, might be used. In such an improvised plant no fractionation of the light oil would probably be feasible. The light oil would be shipped to a larger plant or to a benzol refinery. In a simple plant of this kind the cool wash oil would be pumped from its storage tank to the top of the scrubber. From the bottom of the scrubber the benzolized oil would flow to a storage tank to cool while another portion of benzolized oil was being distilled. After cooling it would be again circulated through the scrubber. A fourth tank would be necessary for the storage of the condensed light oil. If sufficient old materials were available, it might be possible to construct crude heat exchangers, which would improve the economy of the plant. Whether or not a given small plant should attempt to make crude fractions will depend upon local conditions. In general, it does not seem practicable for a plant producing less than 1 800 000 cubic feet of gas per day to do so.

It would hardly be expected that a simple plant of crude construction would recover the benzol as completely or economically as a large well-designed plant, nor is it recommended that a homemade plant be constructed where it is practicable to install a well-designed small plant. However, it is felt that even a crudely designed plant is better than none at all, for a large number of such plants would increase the total benzol and the toluol resources very materially.

In the small plant, as well as in the large one, all reasonable precautions should be taken to make the operation safe, both to the operators and to the plant in general. The highly inflammable character of the materials handled and the explosiveness of the vapors when mixed with air should always be guarded against. The benzol plant should be located where, in case of fire, it will not endanger the rest of the plant. The fact that old equipment is used in the construction should put the operators on the outlook

for leaks, which occur more or less frequently even in well-constructed plants.

20. TIME REQUIRED FOR PLANT INSTALLATION

The time required for the construction of light-oil recovery plants seems to vary greatly. The two essentials to normal construction are sufficient labor to conduct the work in an expeditious manner and the necessary material either at hand or arriving at a steady rate to carry on the work in logical sequence. In this period of the shortage of efficient labor and of the congestion of shops and railroads the period of construction may be prolonged far beyond the normal period, or it may perhaps even be halted. Of course, the amount of construction and the clearing and preparing of the site necessary are large factors in the length of the construction period even in normal times.

If there is no difficulty in obtaining labor and material, a plant to scrub about 10 000 000 feet of gas per day should take approximately three to four months to build. One such plant recovering C. P. products actually took 63 days to build. Another plant recovering crude benzol, toluol, etc., took about four months to build. One plant of homemade construction throughout and scrubbing about three-fourths of a million cubic feet of gas per day, producing crude light oil only, took 35 days to construct and put into operation.

21. MATERIALS USED IN TOLUOL RECOVERY

The materials include wash oil, lubricating oils, packing and repair materials, and in plants producing pure products sulphuric acid and soda are also needed. The wash oil consumption varies considerably in different plants, depending upon the kind of gas washed, the tightness of the circulating system, and the method of operation. While wash oil does not enter into the final product, replacement is necessary at regular intervals, due to losses, depreciation of quality, etc. Some operators replace a certain percentage of wash oil each day, while others replace the whole amount at stated intervals. The percentage of loss varies greatly. Operators claim a replacement of wash oil all the way from 2 to 10 per cent of the number of gallons of light oil recovered. Prices of wash oil at the present time range, according to information available, from 7½ to 12 cents per gallon. These prices are continually changing, so no definite figure can be assigned.

In a pure product plant sulphuric acid and soda are necessary for washing the distillates. The amounts used by various operators differ and are, of course, dependent upon the amount of the various constituents which must be washed out of the product. An average figure seems to be from 0.3 to 0.5 pound of sulphuric acid per gallon of light oil produced and about one-tenth as much soda. Some operators give the amount as 0.8 pound per gallon of the toluol treated.

No figures are available as to the cost of lubricating oils for the numerous pumps, packing, gaskets, etc., but from the nature of the materials handled it seems likely that these expenses are rather heavy.

The consumption of steam, cooling water, and electric power, varying according to local conditions, and the cost of the separate items are, of course, variable. The steam consumption for the stills and accessories alone is estimated by various operators as from 40 to 65 pounds, or even more in some cases, per gallon of light oil produced. The consumption depends largely upon the extent to which the heats of the still effluent and distillate are utilized to heat the incoming benzolized oil. One operator whose plant recovered crude fractions only from 14 000 000 cubic feet of lean by-product oven gas per day stated that his steam consumption was about 65 pounds per gallon of light oil, distributed as follows:

	Pounds
Stripping still	14.2
Superheater	48.8
Crude still	2.0
Total	65.0

In this case there was no vapor-to-oil heat exchanger in use and the oil-to-oil exchanger was an improvised apparatus. The lower figure, 40 pounds per gallon, was quoted by the operating superintendent of a chain of several plants as the requirement of the stills and accessories. No estimate could be obtained of the steam consumption of the various pumps in these plants. In many cases there seems to be no careful record kept of these items. In one plant washing about 12.5 millions cubic feet of mixed gas per day and producing pure products, it was stated that 250 boiler horsepower was required for the entire recovery plant. As this plant produces about 3100 gallons of light oil per day, this would be equivalent to about 57 pounds steam per gallon, assuming that the boiler horsepower used was correctly estimated. One operator

having several plants under his supervision gives 8 to 9 pounds of steam per pound of pure products as an average figure in a plant having two heat exchangers. The question of installing elaborate heat exchangers in a given case to save steam must be decided by local conditions. If the cost of steam production in a plant is very low, it may not be expedient to install all the equipment necessary for the fullest utilization of the waste heat. In a small plant, especially, it might not be feasible to install all this equipment and the use of steam could hardly be expected to be as low per gallon of product as in a larger plant.

The water used in a light-oil recovery plant for cooling purposes is also a very important item. In order to obtain efficient scrubbing of the gas the wash oil must be cooled to 30° C or thereabouts, and the condensers and dephlegmators must have an adequate supply of cooling water or light-oil vapors will be lost. The amount of cooling water used will depend to a great extent upon the temperature of the water supply. A plant which is so fortunate as to have a supply of very cold water will be able to use considerably less than a plant in which the water is relatively warm. A requirement of about 60 gallons of cooling water per gallon of light oil produced seems to be an average amount. One operator of several plants gives 1.2 gallons of water per gallon of wash oil circulated as an approximate figure. A plant which is favorably laid out may find it possible to utilize a portion of the cooling water for other purposes after it has passed through the coolers. Much water may also be saved in some cases by recirculating. The cost of cooling water will in all cases be a controlling factor and will determine how elaborate should be the layout with a view to saving water. Water can be used for cooling in many cases which would not be fit for boiler feed unless treated. In contemplating any light-oil recovery installations, especially in a small plant, one of the first considerations should be the adequacy of the existing steam and water supplies.

PART III.—TOLUOL RECOVERY AND STANDARDS FOR GAS QUALITY [2]

The removal of benzol and toluol from gas necessarily reduces the heating value and candlepower of the gas. The amount of reduction depends upon the quantity of these constituents originally in the gas, the thoroughness of washing, and the general character of the gas with respect to other heating and lighting constituents. In this section it is intended, first, to present a summary of the more important conditions of operation which determine the probable effect of toluol recovery upon the quality of the gas supplied; second, to illustrate the method of estimating the probable effect in any particular case; and, third, to summarize certain general recommendations as to changes in standards that must be made in order that the recovery of toluol can be carried out effectively in a large number of localities. In this section changes of standards are considered only from the standpoint of toluol recovery. No consideration is given to any other factors considered which might properly in many cases make desirable a change of standards. Such matters would depend upon a number of factors not within the scope of the present discussions.

A. SUMMARY OF PRESENT STANDARDS OF GAS QUALITY AND GAS-COMPANY OPERATING CONDITIONS

Both the heating value and the candlepower of gas are used in this country as a measure of the quality of the product supplied. Usually only one of these two characteristics is prescribed by ordinance or administrative ruling, but in some cases both are fixed. In cases where such standards have not been adopted and the quality of the gas supplied is determined by the local gas company, it is of interest to know what quality of gas is being supplied. This information is presented below. For convenience of consideration the companies are classified according to the standard in force. Data are included for all American gas companies making 500 000 000 cubic feet or more of coal, water, or oil gas per year and for such other companies as have been

[2] This section was originally printed in substantially its present form by the Gas Record of Jan. 9, 1918, The Gas Age of Jan. 15, 1918, and the Journal of Industrial and Engineering Chemistry February, 1918. Since its publication several important changes of standards have been made by State and municipal authorities. However, none of these have been indicated in this section, since the summary of standards in force and suggested changes in them are given, primarily, as examples of the treatment of this subject recommended by the Bureau and only secondarily as an indication of the actual standards in force.

recommended to the Ordnance Department for consideration by the subcommittee on coal tar by-products.

1. Gas companies in the following cities are expected to supply gas in compliance with the candlepower requirements, as follows:

(*a*) Requirements of 20 candles or higher —

New York City (including the New York Consolidated Systems, the Brooklyn Union Gas Co., and the Kings County Lighting Co.), 22 cp. Permission has recently been given to change to a heating value of 650 Btu at the same price or to any lower heating value standard if a proportionate reduction in price of gas is made.

Philadelphia, Pa., 22 cp., fixed by a franchise contract with the city.

Des Moines, Iowa, 22 cp.

Sioux City, Iowa, 21 cp.

Omaha, Nebr., 23 cp., measured at the gas works, or 21.2 cp., measured at the city testing station, and 600 Btu heating value.

Charleston, S. C., 20 cp. and 600 Btu.

East St. Louis, Ill., 20 cp. (an old city ordinance requirement) and 565 Btu.

Northern Illinois cities, supplied by Public Service Co., of northern Illinois, 22 cp. (ordinance) and 565 Btu (State standard).

(*b*) Requirements of 18 candles—

Detroit, Mich., 18 cp. and 600 Btu.

Lansing, Mich., 18 cp. and 600 Btu "low value."

Los Angeles, Cal., 18 cp. and 600 Btu. Most of the gas supply of this city is natural gas, which is not limited by these requirements.

(*c*) Requirements of 16 candles—

All cities of Massachusetts, 16 cp., fixed by State statute, but subject to some waiver for purposes of investigation by the State board of gas and electric light commissioners. This board has very recently recommended to the State Legislature a standard of 528 Btu. The following Massachusetts cities are of interest in this connection: Boston, Brockton, Cambridge, Fall River, Haverhill, Lawrence, Lowell, Lynn, Malden, New Bedford, Pittsfield, Springfield, Worcester.

Nashville, Tenn., 16 cp. and 600 Btu.

Jackson, Mich., 16 cp. (20 cp. for water gas) and 600 Btu.

Grand Rapids, Mich., 16 cp. and 600 Btu.

Peoria, Ill., 16 cp. and 565 Btu.

(*d*) Requirements of candlepower less than 16—

Minneapolis, Minn., 15 cp. and 600 Btu.

Birmingham, Ala., 15 cp. and 575 Btu.

St. Paul, Minn., 14 cp. and 600 Btu.

2. Gas companies in the following localities supply gas in compliance with heating value requirements as follows:

(*a*) Total heating value 600 Btu—

St. Louis, Mo.[3]	Washington, D. C.	Atlantic City, N. J.
Baltimore, Md.	Wilmington, Del.	Elizabeth, N. J.
Indianapolis, Ind.	Seattle, Wash.	Jersey City, N. J.
Hammond, Ind.	Tacoma, Wash.	Newark, N. J.
Peru, Ind.	Milwaukee, Wis.	Paterson, N. J.
South Bend, Ind.	Madison, Wis.	Trenton, N. J.
Cedar Rapids, Iowa.		

[3] A municipal requirement; the State requirements is 570 Btu.

(b) Total heating values 585 and below—

Denver, Colo., 575.
Bridgeport, Conn., 575.
Hartford, Conn., 575.
New Haven, Conn., 575.
Waterbury, Conn., 575.
Portland, Oreg., 570.
Ardmore, Pa., 570.
Allentown, Pa., 570.
Chester, Pa., 570.
Reading, Pa., 570.
Wilkes-Barre, Pa., 570.
Manchester, N. H., 565.
Chicago, Ill., 565.
San Francisco, Cal., 550.
San Diego, Cal., 550.
Oakland, Cal., 550.
Cities of New York State, 585. Of these cities the following are of interest in this connection: Albany, Binghamton, Buffalo, Poughkeepsie, Rochester, Schenectady, Syracuse, Troy, and Utica.

3. The gas companies in the following cities have no requirements limiting the candlepower or heating value of the gas which they supply but are reported to be supplying gas of candlepower and heating value as given below:

New Orleans, La., 22 cp. and 600 Btu.
Jacksonville, Fla., 20 cp. and 580 Btu.
Atlanta, Ga., 19 cp. and 600 Btu.
Richmond, Va., 18 cp. and 590 Btu.
Pawtucket, R. I., 17 cp. amd 600 Btu.
Providence, R. I., 17 cp. and 600 Btu.
Salt Lake City, 17 cp. and 600 Btu.
Houston, Tex., 17 cp. and 585 Btu.
Mobile, Ala., 15 cp. and 600 Btu.
Portland, Me., 15 cp. and 570 Btu.
Savannah, Ga., 575 Btu.
Battle Creek, Mich.
San Antonio, Tex.

B. METHOD OF ESTIMATING INFLUENCE OF TOLUOL RECOVERY UPON GAS QUALITY

As previously stated the quantity of toluol or benzol in the gas initially is a large factor in determining the quality of the gas both before and after removal of the toluol, since the conditions which make for the presence of large quantities of these aromatic hydrocarbons are the conditions prevailing during the production of high candlepower and high-heating-value gases. In general the quantity of toluol and other light oils present in water gas depends upon the amount of gas oil used in the production of this gas. Approximately 10 per cent of the volume of gas oil used can be recovered as crude light oil, and of this amount from one-fifth to one-sixth can be recovered as pure toluol. Coal gas made by any of the usual horizontal retort processes, which are the only processes of coal-gas manufacture requiring particular consideration in this report, usually contains about one-fourth to one-third of a gallon of light oil per 1000 cubic feet, depending upon the character and treatment of the coal and the quality of the gas. From one-eighth to one-tenth of this light oil is recoverable as pure toluol.

From each one-tenth gallon of light oil removed per 1000 cubic feet of gas the total heating value is reduced by approximately 10

to 14 Btu per cubic foot and the candlepower by 2½ to 3 candles. However, restoring part of the light oil removed, for example, enriching with the benzol fraction, may in some measure compensate for the loss in heating value and candlepower brought about by the initial washing. In fact, if a sufficient amount of additional benzol is available the candlepower and heating value can be restored substantially to the original values. The increase in quality is about the same per unit of volume of benzol returned as was the loss on removal of the light oil. However, this practice would not generally be practicable since it demands the purchase of benzol or other enriching constituents to take the place of those constituents which are permanently removed from the gas. In estimating the loss of candlepower and heating value, the figures here presented are probably slightly higher than would correspond to the change in quality of gas at the customers since in distributing unwashed gas there is usually considerable loss due to condensation.

From these two generalizations and a knowledge of the initial candlepower and heating value of the gas, it is readily possible to estimate approximately the influence upon the quality of the gas of recovering different amounts of toluol or of toluol and benzol. Such estimates are, of course, not exact, but they furnish an excellent guide for readjustment of standards in any case where this is necessary or for approximating the quantity of materials which can be obtained by washing the gas. The following examples will make clear the application of the data:

Example 1.—Assume water gas made from 4 gallons of oil per 1000 cubic feet and having an open-flame candlepower of 20 and a heating value of 625 Btu. About 0.4 of a gallon of light oils per 1000 cubic feet could be recovered from such gas with practically complete washing. If none of the benzol was returned to the gas, the result would be a gas of about 575 Btu and 10 candlepower. If the light oil were distilled and the benzol fraction were returned to the gas, the loss in heating value and candlepower would be perhaps one-half as great and the result a gas of about 600 Btu and 15 candles. In order to restore the heating value and candlepower to substantially their original figures, it would be necessary to add benzol to the extent of approximately 0.2 gallon per 1000 cubic feet of gas manufactured. From the 0.4 gallon of light oil originally obtained some 0.07 to 0.08 gallon of toluol would probably be obtainable on refining.

Example 2.—Assume carbureted water gas with 3 gallons of gas oil per 1000 cubic feet and assume a very high oil efficiency so that the candlepower was 18 and the heating value 570 Btu. From this gas about 0.3 gallon of light oil, equivalent to perhaps 0.05 to 0.06 gallon toluol, would be obtained per 1000 cubic feet with commercially complete washing. The result of this washing would be a gas approximately 530 Btu and 9 to 10 candles, which would be restored to 550 Btu and about 12 candles if reenriched with the benzol portion of the light oil.

Example 3.—A mixture of coal gas and water gas in about equal proportion may be assumed made from water gas for which 3½ gallons of gas oil were used to give 18 candles and 600 Btu and coal gas of 580 Btu and 15 candles. Such mixed gas would yield perhaps 0.3 gallon light oil per 1000 cubic feet, and the average candlepower would be reduced by washing from 16½ to 8 or 9 candles and the heating value from 590 Btu to about 550 Btu. Restoration of the benzol fraction would give a product of about 12 candles and 570 Btu.

Example 4.—A coal gas made from ordinary grade of gas coal to yield 10 000 cubic feet of gas per ton is assumed to produce gas of about 14 candles in the open flame and of 585 Btu. From this approximately 0.3 gallon of light oil per 1000 cubic feet of gas could be recovered and from it 0.025 to 0.03 gallon of toluol per 1000 cubic feet would be available. The gas after washing would have approximately 8 candles and 550 Btu, which would be increased to perhaps 12 candles and 570 Btu if the benzol fraction was restored.

In any of the above cases the net loss in heating value and candlepower might readily be reduced if some of the other constituents of the light oils such as the solvent naphtha fraction were also restored to the gas or the loss in heating value and candlepower could be made less by operating the washing equipment in such a way as to accomplish only a partial removal of the light oils. In the latter case if removal of only 75 per cent of the quantity of light oil readily obtainable were considered satisfactory this would make the losses in heating value and candlepower of only about three-fourths as great as above indicated, but of course it would also somewhat reduce the yield of toluol.

It is probable that complete washing of the gas with restoration of the benzol in most cases will be considered advisable, since the need for toluol is considerable and very little sacrifice of toluol yield can be allowed. But the demand for benzol is not so great

and the restoration of the benzol to the gas might give results at first more satisfactory to the gas users than would the sale of this benzol, with the slight reduction in total costs for the gas which might possibly be accomplished thereby. Especially might the restoration of benzol be necessary where a high candlepower standard has been in force, since otherwise the loss in candlepower would be rather greater than would be desirable at one time. In any computation, therefore, it is probably best to assume, unless other basis is known to be correct, that commercially complete washing of the gas would be necessary and that the benzol fraction of the light oil, amounting to approximately one-half the total volume of light oil removed, will be restored to the gas.

C. RECOMMENDATIONS REGARDING STANDARDS FOR GAS QUALITY

From the estimates in the preceding section it is evident that much greater difficulty is met in complying with a candlepower requirement after removal of toluol or light oil than is encountered if a heating value standard is to be complied with. Because of this fact it seems desirable that in any case where toluol is to be removed the candlepower standard be altogether eliminated or be made sufficiently low so that it will not interfere seriously with the proposed operations. Many other factors independent of toluol recovery make evident the desirability of eliminating candlepower requirements and substituting heating value requirements as the primary basis of gas measurement. Therefore, the war is only an added influence tending to hasten an end otherwise desirable.

In all cases where the candlepower has previously been below 18 it would seem that the elimination of the candlepower requirement altogether would be reasonable, although in any event it is expected that the company would supply a gas of at least 8 or 10 candles, which would be sufficient to care for the need of those customers who must use some portion of the gas for open-flame lighting. In cases where 18 or 20 candles or higher has been maintained regularly in the past, it might be undesirable to have the candlepower go below 12 to 14, unless open-flame lights were generally eliminated and a readjustment of the appliances of all customers were made wherever the change in quality might make this necessary. For all companies which have geen complying with requirements of 18 candlepower or higher, an understanding might be reached as to the maintenance of at least 12 to 14 candles

for such a period as might be necessary to accomplish a general adjustment to the new conditions.

When coal gas is supplied either alone or mixed with very small percentages of water gas, it is impracticable to make a very rich gas, since the character of the coals available in most instances would preclude economic operation if a higher standard, either of heating value or of candlepower must be maintained. For cities where only coal gas is supplied the standard could scarcely be higher than about 570 Btu if practically complete toluol recovery is expected. Higher heating value standards than this would probably have to be modified for such gas supplies.

If water gas is manufactured, either alone or as a major constituent of the supply, it is entirely practicable to make a gas of reasonably high heating value and candlepower initially and have after removal of the toluol a heating value of 585 to 600 Btu. In each case it would be a question as to which procedure was the more economical; that is, whether it would be better to make the same quality of gas as had previously been supplied and supply the customer with a somewhat lower product than formerly after the toluol had been removed from the gas, or to make the gas initially somewhat richer than before by the use of slightly more gas oil per 1000 cubic feet, so that the product after washing would have substantially the same heating value as had previously been supplied. If the quality previously supplied was rather high, approaching the maximum of the range of quality permissible for efficient operation, then any increase in the initial quality would obviously be undesirable; but otherwise an initial increase in quality with subsequent washing down to the original might be the best practice. Since the quantity of toluol available is largely dependent upon the initial richness of the gas which has been washed, there is considerable advantage from the standpoint of the Government in having the richest practicable gas made initially; but, of course, in any case the limits of economical operation must be clearly recognized, and conservation of oil might also be an important factor.

As a summary of these points the following suggestions are offered as desirable adjustments to facilitate the recovery of toluol:

1. Eliminate all candlepower requirements now in force, except for the cities where 18 candles or higher has been supplied, in which localities reach an understanding that at least 12 candles will be maintained for a period, say a year, during which time re-

adjustments of appliances and substitution of mantle lamps would be accomplished to such an extent as to justify complete elimination of candlepower regulations.

2. For plants making coal gas (or practically only coal gas) let the heating value standard be from 550 to 570 Btu.

3. For plants making water gas, either alone or as a major constituent, let the heating value standard be 570 to 600 Btu monthly average total heating value, the adjustment being made between these limits according to the economic conditions of operation.

In order to show the number of companies that would be affected by these several recommendations, the following tabulation of the companies above listed is prepared. This tabulation does not take account of any unusual local conditions which might affect some of the cases materially. The kind of gas manufactured is also indicated: W = water gas, C = coal gas, O = oil gas, M = mixed coal and water gas, B = by-product coke-oven gas, and N = natural gas.

1. Localities in which no change of standard will probably be needed and no serious change in the quality of gas supplied will probably result:

Chester, Pa., −W+B.
Reading, Pa., −W.
Wilkes-Barre, Pa., −W.
Ardmore, Pa., −W.
Allentown, Pa., −W.
Portland, Oreg., −O.
Manchester, N. H., −M.
Hartford, Conn., −M.
Bridgeport, Conn., −W.
New Haven, Conn., −M.
Waterbury, Conn., −W.
Denver, Colo., −M.
San Antonio, Tex., −W.
Savannah, Ga., −W.
Jacksonville, Fla., −M.
Richmond, Va., −M.
Atlanta, Ga., −M.
New Orleans, La., −W.
Albany, N. Y., −W.
Schenectady, N. Y., −M.
Troy, N. Y., −W.
Utica, N. Y., −W.
Poughkeepsie, N. Y., −W.
Syracuse, N. Y., −M.
Binghamton, N. Y., −W.
Rochester, N. Y., −M.
Chicago, Ill., −W−B−N.
San Diego, Cal., −W+O.
San Francisco, Cal., −O.
Oakland, Cal., −O.
Houston, Tex., −W.
Pawtucket, R. I., −M.
Providence, R. I., −M.
Battle Creek, Mich., −M.
Portland, Me., −M.
Salt Lake City, Utah, −M.
Mobile, Ala., −M.

2. Localities in which a candlepower standard may have to be abandoned, but with no serious change in the heating value of the gas supplied:

Lynn, Mass., −M.
Boston, Mass., −M.
Brockton, Mass., −M.
Lawrence, Mass., −M.
Lowell, Mass., −M.
New Bedford, Mass., −M.
Pittsfield, Mass., −M.
Worcester, Mass., −M.
Cambridge, Mass., −M.
Fall River, Mass., −M.
Haverhill, Mass., −W.
Springfield, Mass., −M.
Malden, Mass., −M.
Birmingham, Ala., −M.
Waterloo, Iowa, −W.
Peoria, Ill., −M.
Nashville, Tenn., −M.
East St. Louis, Ill., −W+N.

3. Localities in which slight change in heating value regulations may perhaps be required, but in no case probably more than equivalent to 5 per cent of the present value. The six cities marked (*) have candlepower standards which should be eliminated also.

Indianapolis, Ind., B+W.
Tacoma, Wash., C+O.
Seattle, Wash., B+M.
Trenton, N. J., B+N.
Paterson, N. J., −W.
Newark, N. J., −M.
Jersey City, N. J., −W.
Elizabeth, N. J., −W.
Atlantic City, N. J., −W.
Washington, D. C., −M.
*St. Paul, Minn., −W.
*Minneapolis, Minn., −M.
*Detroit, Mich., −M+B.
Buffalo, N. Y., −M.
Milwaukee, Wis., −M.
Madison, Wis., −W.
Cedar Rapids, Iowa, −M.
St. Louis, Mo., M+B.
Baltimore, Md., −W+B.
Hammond, Ind., −M.
South Bend, Ind., −M.
Peru, Ind., −M.
Wilmington, Del., −W.
*Grand Rapids, Mich., −M.
*Jackson, Mich., −M.
*Los Angeles, Cal., −O+N.

4. Localities in which high candlepower regulations should be changed or eliminated in order to permit operation on a heating value basis; the reduction in heating value of the gas delivered would probably be a considerable percentage of the present value. In the cases of Lansing and Omaha a lower heating value than now in force would also be necessary.

New York City, −M and W.
Omaha, Nebr., −W.
Lansing, Mich., −M.
Philadelphia, Pa., −M.
Charleston, S. C., −W.
Des Moines, Iowa, −W.
Sioux City, Iowa., −W.
Northern Illinois cities, supplied by Public Service Company of Northern Illinois, −M.

PART IV.—FORM OF NET COST CONTRACT FOR OPERATION OF TOLUOL AND LIGHT OIL RECOVERY PLANT

The following is the form contract used by the Ordnance Department with gas companies as the basis for operation of Government-owned toluol plants at city gas works:

This Agreement, entered into this ——— day of ———, 1918, between ——— Company, a corporation organized and existing under and by virtue of the laws of the State of ———, having its principal office at ———, in said State (hereinafter called the Contractor), of the first part, and the UNITED STATES OF AMERICA, by Samuel McRoberts, Colonel, Ordnance Department, National Army (hereinafter called the Contracting Officer), acting by and under authority of the Chief of Ordnance, United States Army, and under the direction of the Secretary of War, of the second part:

Witnesseth,

WHEREAS the Contractor owns and operates ——— gas plant— at ——— (hereinafter called the "Gas Works"), for the purpose of supplying gas for public consumption, with a rated daily capacity of ——— cubic feet per day of ——— gas; and

WHEREAS a state of war exists between the United States of America and the German and Austro-Hungarian Governments, constititing a national emergency; and

WHEREAS it is possible to recover from the gas supplied by the Contractor light oils containing toluol and such toluol is necessary for the prosecution of the said war; and

WHEREAS there will be required for this purpose special buildings and apparatus to be used in connection with the equipment of the Contractor; and

WHEREAS the Contractor has given the United States permission, and the United States has undertaken, to provide and erect upon the property of the Contractor additional buildings, equipment, machinery, appliances, and other facilities required for the recovery of light oils containing toluol from the gas supplied by the Contractor; and

WHEREAS, pursuant to the direction of the United States, the Contractor will maintain and operate said additional buildings, equipment, machinery, appliances, and other facilities now or hereafter to be provided by the United States (hereinafter called the "Increased Facilities") at the actual net cost of so doing, and the United States intends to reimburse the Contractor for such cost, so that the Contractor will not sustain any loss as the result of the performance of this contract on its part:

NOW, THEREFORE, under the laws of the United States and the Executive orders of the President of the United States or heads of its departments in such cases made and provided, and in consideration of the mutual agreements herein contained, the said parties have agreed, and by these presents do agree to and with each other, as follows, viz:

ARTICLE I. The United States shall provide and erect at its own expense upon the property of the Contractor such Increased Facilities as will, in addition to the Contractor's facilities, enable the Contractor to recover light oils containing toluol and to comply with all requirements of this contract in a manner satisfactory to the Contracting Officer. The Contractor hereby consents to the entry, use, and occupation by the United States or its contractors upon and of the premises for the construction and operation of the Increased Facilities.

The Increased Facilities shall be and remain the property of the United States until removed, disposed of, or sold to the Contractor as hereinafter provided, and shall, so far as practicable, be kept separate and apart from property belonging to the Contractor. The Contractor shall not acquire any property right, title, or interest of any kind in the Increased Facilities. The Increased Facilities shall be at all times considered personalty, which shall not be deemed to be affixed to or in any way part of the real estate to which said Increased Facilities are attached.

The Contracting Officer may, at his option, from time to time, without expense to the Contractor, rebuild, alter, enlarge, or otherwise modify the Increased Facilities.

ARTICLE II. In so far as the Contractor can legally obligate itself, the Increased Facilities may be removed without expense to the Contractor by the United States at any time upon thirty (30) days' notice to the Contractor. The Increased Facilities shall be removed by the United States without expense to the Contractor within one (1) year from the termination of the war between the United States and the German Government; provided, however, that the Contractor shall (in so far as the laws of the United States may allow) have the option within ninety (90) days from said date, or from the date of the termination of this agreement as provided in Article XI hereof (during which ninety (90) days no part of the Increased Facilities shall be removed), of purchasing all or part of the Increased Facilities at a price to be mutually agreed upon by the Contracting Officer and the Contractor. In the event of removal by the United States of the Increased Facilities, the United States shall place the property affected by the construction of the Increased Facilities in as good condition as obtained prior to the construction of the Increased Facilities, subject only to reasonable wear and tear, all without expense to the Contractor. The United States shall reimburse the Contractor for any expense actually and necessarily caused by such removal. Such removal shall not interrupt the manufacture and distribution of gas. If the Increased Facilities located upon the property of the Contractor shall not have been removed by the United Stqtes within the time above prescribed, or within such further reasonable period not to exceed six (6) months additional, as may be prescribed in writing by the Contracting Officer, the Increased Facilities shall be removed by the Contractor at the cost of the United States.

ARTICLE III. The Contractor hereby agrees:

(a) That it will permit, aid, and facilitate in every way the prompt construction, maintenance, and operation of the Increased Facilities;

(b) That it will maintain and operate with all due diligence and efficiency the Increased Facilities and the Gas Works, so far as the operation of the Gas Works may affect the recovery and treatment of the light oils containing toluol, all as directed by the Contracting Officer in writing from time to time, the right being reserved to the Contracting Officer to limit the quantity of light oil or of any component thereof to be obtained and to stop operation of the Increased Facilities entirely at any time upon five (5) days' notice: *Provided, however,* That if the Contractor be required hereunder or by direction of the Contracting Officer to perform any act claimed to be in violation of or inconsistent with the laws, regulations, and ordinances of any local, State, or Federal authority or the provisions of any contract heretofore made by the Contractor, the United States shall afford the Contractor full and complete protection in the carrying out of the terms of this contract and of the directions of the Contracting Officer. The Contractor shall promptly notify the Contracting Officer of every proceeding brought against the Contractor for violation of any such statute, regulation, or ordinance in which the Contractor may claim that said violation is due to its compliance with the terms of this contract or the directions of the Contracting Officer. *And provided further,* That no order or direction of the Contracting Officer shall be deemed to require or authorize the Contractor to interrupt the manufacture or supply of gas.

(c) That it will begin said operation and maintenance in part or in whole as directed within fifteen (15) days from the date of written notice to that effect from the Contracting Officer.

(d) That it will treat or permit to be treated all gas manufactured at the Gas Works, as the Contracting Officer may direct, for the recovery of the light oils containing toluol; that it will not, except in case of necessity, interfere with or reduce the normal and usual amount of gas manufactured at the Gas Works; and that it will furnish said gas to the Increased Facilities at as low a temperature as the Contractor's facilities will permit.

(e) That it will promptly pay for all materials, supplies, tools, appliances, repair parts, labor, and other services rendered and required for the performance of this contract which are provided by the Contractor.

The Contractor shall not be held responsible for delays due to the fault of the United States, nor for delays due to riots, labor strikes, acts of war, fires, and other causes beyond the control of the Contractor; but immediately after the removal of the cause of such delay the Contractor shall proceed with the performance of this contract, due allowance for such delay having been made, and the Contractor agrees, in view of the emergency necessitating this contract, to use its best efforts to remove such causes of delay.

The Contractor shall not, without the consent in writing of the Contracting Officer, enter into any arrangement or contract affecting in any manner the recovery, treatment, sale, or disposal of crude or pure toluol, or of any other component of the light oils obtained by the operation of the Increased Facilities.

In operating, caring for, and storing the property of the United States, the Contractor shall use its best efforts to protect the same, but it shall not be liable for any loss or damage thereto except such as may be caused by its willful fault or by such neglect as is not excusable.

The United States hereby agrees, for and during the term of this agreement, to indemnify and hold the Contractor harmless from any and all costs, charges, and expenses (including liability for damages) imposed upon the Contractor by the municipal authorities in any permit for or consent to the construction, existence, use, maintenance, operation, or removal of any mains, pipes, conduits, poles, or any other sub or super structures in the streets which form part of the Increased Facilities.

ARTICLE IV. The Contractor shall, within fifteen (15) days from the date of written notice from the Contracting Officer, provide with the utmost dispatch and thereafter maintain such administrative, purchasing, accounting, engineering and chemical organization and such labor and such materials, supplies, tools, appliances, repair parts, and the like, as may be necessary for the efficient operation of the Increased Facilities as provided herein and for compliance with all the requirements of this contract in a manner satisfactory to the Contracting Officer.

All materials, supplies, tools, appliances, repair parts, and the like paid for or required herein to be paid for by the United States shall be and become the property of the United States immediately upon delivery at the site of the Increased Facilities. All crude toluol obtained in the operation of the Increased Facilities shall be and become the property of the United States. All other products obtained in the operation of the Increased Facilities shall, if so ordered in writing by the Contracting Officer, be and become the property of the United States or shall be used or disposed of by the Contractor as may be directed by the Contracting Officer.

ARTICLE V. Until otherwise directed by the Contracting Officer, the crude toluol produced by the Contractor shall, except for causes beyond its control, contain at least seventy (70) per cent of pure toluol and a proportion of paraffin not greater than one and one-half per cent (1½%) of the pure toluol content. The determination of pure toluol and paraffin content shall be in accordance with "Laboratory Standards to be used in connection with light oil recovery plants" as prescribed by the Chief of Ordnance.

ARTICLE VI. The Inspecting and Receiving Officer designated by the Contracting Officer shall test the crude toluol produced, utilizing the facilities of the Gas Plant to whatever extent necessary, and shall accept or reject in writing the toluol so tested. If any crude toluol is rejected, the Contractor shall retreat the same as directed.

The Contractor shall load the crude toluol in suitable containers furnished by the Contracting Officer, marked as directed by the Contracting Officer, and shall ship or store the same for as long a period as the Contracting Officer may deem necessary.

If storage facilities be required in addition to those provided by the Increased Facilities, the Contractor shall use its best efforts to arrange for such additional storage facilities, and shall utilize them as directed by the Contracting Officer.

ARTICLE VII. The United States shall pay to the Contractor the net cost actually and necessarily incurred by the Contractor for the operation and maintenance of the Increased Facilities, said cost being:

(a) The salaries and wages of all persons necessarily and directly employed. In the operation of the Increased Facilities, the Contractor shall make no departure, without the written consent and approval of the Contracting Officer, from the standard of wages being paid where said work is being done.

(b) The cost of all necessary materials, supplies, tools, appliances, repair parts, and the like, except as hereinafter provided.

(c) ——— dollars ($———) for each one thousand (1,000) cubic feet of water actually and necessarily purchased for and supplied to the Increased Facilities by the Contractor; and ——— dollars ($———) for each one thousand cubic feet of other water actually and necessarily pumped by the Contractor for the Increased Facilities: *Provided, however,* That if the price for steam as hereinafter fixed in paragraph (d) hereof shall be changed as provided for therein, then the price for water pumped may be correspondingly changed to a new price which shall be as mutually agreed upon by the Contracting Officer and the Contractor, or, if an agreement is impossible, as may be fixed by the Chief of Ordnance. Water shall be measured by meter at the site of the Increased Facilities.

(d) ——— cents (—— c.) for each thousand pounds of dry steam actually and necessarily supplied during the first three months of the operation of the Increased Facilities and thereafter as mutually agreed upon by the Contracting Officer and the Contractor, or, if an agreement is impossible, as may be fixed by the Chief of Ordnance.

Steam shall be furnished by the Contractor at a guage pressure of not less than one hundred (100) pounds and shall be measured by meter at the site of the Increased Facilities.

(e) The cost of additional enriching oil. By the phrase "additional enriching oil" is meant the enriching oil, if any, ordered used by the Contracting Officer, which is in excess of the amount of enriching oil used (per each one thousand (1,000) cubic feet of carburetted water gas made as measured by the Gas Works station meters corrected to 60° Fahr.) by the Contractor in the operation of the Gas Works during the years ——— to ———, inclusive, which it states to be:

SCHEDULE

December	——— gallons	June	——— gallons
January	——— gallons	July	——— gallons
February	——— gallons	August	——— gallons
March	——— gallons	September	——— gallons
April	——— gallons	October	——— gallons
May	——— gallons	November	——— gallons

The Contractor covenants and agrees that it will use an amount of enriching oil in the operation of the Gas Works equal, month by month, to the amounts above stated for the respective months, provided that its supply of enriching oil is not curtailed by causes beyond its control.

Provided further, however, That if it be finally determined by any tribunal or court of competent jurisdiction, in any proceeding to fix the just and reasonable rates to be paid for gas, that any of the enriching oil set forth in the above schedule should be disallowed in fixing such rate, because such oil so disallowed is absorbed by the Increased Facilities, then the United States shall compensate the Contractor for the cost of such enriching oil so disallowed as though it were additional enriching oil.

The cost of the net loss in volume of gas because of the operation of the Increased Facilities at the rate of forty cubic feet of gas for each gallon of toluol, benzol, and solvent naphtha recovered by the operation of the Increased Facilities and not restored to the gas at the outlet of the Increased Facilities.

The cost of enriching oil shall be the actual net cost to the Contractor of the enriching oil in the tanks of the Contractor at the Gas Works during the month in question: *Provided, however*, That if, at the termination of the present contract or contracts for the purchase of enriching oil by the Contractor, the cost of enriching oil thereafter paid by the Contractor shall be greater or less than the cost paid by it under the present contract or contracts, a proper adjustment for this difference in cost shall be made to cover the amount of additional enriching oil used prior to the termination of said contract or contracts.

The provisions of this subdivision (e) of this article shall continue for the first three months of the operation of the Increased Facilities and thereafter as may be mutually agreed upon by the Contracting Officer and the Contractor, or, if an agreement is impossible, as may be fixed by the Chief of Ordnance.

(f) The rental, at the customary rates where the Increased Facilities are being operated, of necessary facilities and equipment rented.

(g) The reasonable rental for property (exclusive of the land indicated on the annexed blue prints) owned by the Contractor and actually and necessarily used as part of the light oil recovery plant, such rental to be as mutually agreed upon by the Contracting Officer and the Contractor, or if agreement is impossible, as may be fixed by the Chief of Ordnance. If said property, or any of it, is not used exclusively or for the entire time for the purposes of this contract, the rental shall be prorated according to the amount of space or time for which said property is used. Said rental, except in the case of machinery and apparatus, shall in no event exceed ten (10) per cent per annum of the cost of said property, or of the part thereof so used. Provided, however, that no rental shall be paid for any property used for or in connection with the production and supply of water or steam, except such rental as is included in the prices for water and steam in paragraphs (c) and (d) of this Article.

(h) The premiums on such bonds, fire, liability, and other insurance as the Contracting Officer may approve or require or as are required by the provisions of Articles X and XIX, including the premiums paid by the Contractor for insurance on the Gas Works against the extra hazard attributed to the maintenance and operation of the Increased Facilities in connection therewith.

(i) Permit fees and other similar items of expense.

(j) An amount equal to the actual and necessary loss incurred by the Contractor because of the reduction in the quantity of drip oil recovered. Said loss shall be computed for the three months ending March 31, June 30, September 30, and December 31, respectively; and it shall be determined by multiplying (1) the average price actually received by the Contractor per gallon of drip oil during the quarter, by (2) the difference between the quantity of drip oil obtained during the quarter and that obtained during the corresponding quarter of the calendar year 1917.

(k) Such other items, credits, or allowances (including any unusual or extraordinary expenditures incurred in complying with the provisions of this agreement or in connection therewith) as should in the opinion of the Contracting Officer be paid, allowed, or included. When such an item is allowed by the Contracting Officer it shall be specially certified as being allowed under this subdivision.

The Contractor shall credit against the cost hereinbefore stated the net amount received from the sale or disposal of any materials, supplies, appliances, and the like, and of anything produced by or obtained from the operation or maintenance of the Increased Facilities.

The Contractor shall take advantage to the extent of its ability of all prices on materials for the United States which are lower than the prices at which the Contractor can secure such materials unless so doing will delay the operation of the Increased Facilities. In every such case the Contractor shall notify the Contracting Officer of its inability to purchase such material at the prices paid by the United States.

The United States reserves the right to supply any materials to be used for the operation or maintenance of the Increased Facilities, and to pay to common carriers any or all freight charges on materials and supplies of all kinds furnished by the Contractor and certified by the Contracting Officer as being for the purpose of this contract.

As soon as practicable after the first day of each month the Contractor shall submit to the Contracting Officer a statement of the materials shipped or stored on order of the Contracting Officer during the preceding month and of the net amount due the Contractor as hereinbefore provided, and payment shall be made upon the certificate of the Contracting Officer as soon as practicable thereafter.

ARTICLE VIII. The Contractor shall, from time to time and whenever requested by the Contracting Officer, furnish to the Contracting Officer full and complete statements and reports of the operation and maintenance of the Increased Facilities and the Gas Works upon any factor affecting this contract. The Contractor shall follow any instructions relating to said statements and reports which the Contracting Officer may from time to time give, including instructions as to:

(1) Books of accounts kept for the purposes of this contract;

(2) The submission of statements, bills, and other supporting papers;

(3) The submission of engineers' and accountants' certificates;

(4) Such regulations and instructions with regard to the determination of cost as from time to time shall be adopted by the Chief of Ordnance or as may be required in order to enable the Contracting Officer to issue the proper certificates for payment thereof; and

(5) The keeping of records, and the preparation and submission of reports to the Contracting Officer.

ARTICLE IX. All crude toluol and other products obtained pursuant to this contract, the plant, machinery, tools, equipment, materials, supplies, all workmanship, and all bills, statements, receipts, books, vouchers, correspondence, and all other records of all sorts of the Contractor in any way related to this contract shall be at all times subject to inspection by the officers and agents of the Ordnance Department or of the Contracting Officer; and the Contractor shall furnish reasonable facilities and assistance for all such inspection. The Contractor shall keep all such records convenient for ready reference and shall preserve the same for a period of at least six (6) years after the completion of this contract, unless further compliance with this provision is waived by the Contracting Officer subsequent to the completion of this contract.

ARTICLE X. The Contractor shall, at the cost of the United States (or if it insures itself under the ——— Law of the State of ——— it shall be entitled to reimbursement at the current rates of insurance of similar character) insure its liability to its employees. The Contractor shall take out and carry such other insurance as may be required in writing by the Contracting Officer. All insurance shall be procured and renewed by the Contractor and shall continue during the period of this agreement.

ARTICLE XI. The term of this contract shall be from the date hereof until one (1) year from the termination of the war between the United States and the German Government, provided, however, that the Contracting Officer may at his option cancel

and terminate this agreement at any time upon fifteen (15) days' notice in writing to the Contractor. In the event of such cancellation and termination of this contract, the United States shall pay to the Contractor all costs to date allowed and determined and not previously paid and also all net outstanding obligations of the Contractor actually and necessarily incurred for the performance of this contract and from which the Contractor can not otherwise be relieved: or the United States may, at its option, assume such obligations.

ARTICLE XII. In the event of failure of the Contractor to comply with the terms of this contract or any of them, this contract may be terminated by notice in writing to the Contractor without prejudice to any claim the United States may have against the Contractor. Thereupon the Contracting Officer may proceed to recover the light oils containing toluol from the gas manufactured at the Gas Works, and may employ such agents and employees, including the Contractor, its agents and employees, as the Contracting Officer may deem necessary or desirable, and may take possession of the Increased Facilities, may secure from the Contractor an assignment of any contract or agreement relating to this contract, which said contract or agreement the Contractor hereby agrees to assign, and may generally do and perform all acts and things necessary or convenient in order to obtain the materials which are the subject of this contract.

In the event of the termination of this contract as aforesaid, the United States shall pay to the Contractor all costs to date, allowed and determined and not previously paid, and also all net outstanding obligations of the Contractor actually and necessarily incurred for the performance of this contract and from which the Contractor can not otherwise be relieved, or the United States may, at its option, assume such obligations.

ARTICLE XIII. The Contractor hereby, for the consideration herein named, waives and releases all lien or right of lien now existing or that may hereafter arise for work or labor performed or materials furnished or for any other reason or cause under this contract, under any lien law, State or Federal, upon any Increased Facilities, material, supplies, and the like, coming into its possession which it is herein contemplated shall presently or ultimately become the property of the United States; and the Contractor agrees not to create or suffer to be created any mortgage, lien, pledge, attachment, or other incumbrances upon any such Increased Facilities, material, supplies, or other such property in its possession, and in the event that such mortgage, pledge, lien, attachment, or other incumbrance is created the Contractor agrees to pay and discharge the same, or if it disputes the validity of the claim out of which such incumbrance arises, immediately to bond the same, to the end that all such property shall at all times be and remain free from all incumbrances.

ARTICLE XIV. This contract shall not, nor shall any right to receive payment or any other interest therein, be transferred or assigned by the Contractor to any person, firm, or corporation. In the event of insolvency, receivership, bankruptcy, or other proceedings of or against the Contractor, the Contracting Officer may, in his discretion, terminate this agreement and assume and undertake the operation of the Increased Facilities.

The Contractor shall make all subcontracts, purchases, payments, and arrangements for performing this contract in its own name and for its own account, and shall not bind or purport to bind the United States, except as the Contracting Officer shall otherwise direct in writing. All subcontracts must be approved by the Contracting Officer.

The Contractor shall, unless otherwise directed by the Contracting Officer, insert in every contract hereafter made for facilities, labor, material, supplies, and the like, or otherwise necessary to the performance of this contract a provision that such contract may be assigned by the Contractor and that it relates to a "main contract" between the Contractor and the United States.

ARTICLE XV. No Member of or Delegate to Congress or Resident Commissioner is or shall be admitted to any share or part of this contract, or to any benefit that may arise therefrom, but this Article shall not apply to this contract so far as it may be within the operation or exception of section 116 of the Act of Congress approved March 4, 1909 (35 Stats., 1109).

ARTICLE XVI. No person or persons shall be employed in the performance of this contract who are undergoing sentences of imprisonment at hard labor which have been imposed by the courts of the several States, Territories, or municipalities having criminal jurisdiction.

ARTICLE XVII. Any doubts or disputes which may arise as to the meaning of anything in this contract shall be referred to the Chief of Ordnance for determination. If, however, the Contractor shall feel aggrieved at any decision of the Chief of Ordnance upon such reference he shall have the right to submit the same to the Secretary of War, whose decision shall be final.

ARTICLE XVIII. When notice under this contract is not actually delivered at the office of the Contracting Officer or of the Contractor it shall be deemed to have sufficiently been given to and received by the Contracting Officer or the Contractor as the case may be, when mailed in a sealed postpaid wrapper to the office of the Contracting Officer or to the principal office of the Contractor at the address above written.

ARTICLE XIX. The Contracting Officer may at any time demand that the Contractor furnish a bond in such sum as he may fix for the faithful performance of this contract with sureties satisfactory to the Contracting Officer. If the Chief of Ordnance shall at any time deem such sureties to be unsatisfactory, and he shall so notify the Contractor, the latter shall forthwith submit in substitution new sureties acceptable to the Chief of Ordnance. For failure on the part of the Contractor promptly to comply with such demands, the Chief of Ordnance may by written notice to the Contractor terminate the contract.

ARTICLE XX. This contract may be executed in any number of counterparts, all of which shall constitute one original contract. Wherever the words "Contracting Officer" are used herein, the same shall be construed to include his successor or successors, his duly authorized agent or agents, or anyone from time to time designated by the Chief of Ordnance to act as Contracting Officer.

WASHINGTON, August 1, 1918.

DEPARTMENT OF COMMERCE

TECHNOLOGIC PAPERS

OF THE

BUREAU OF STANDARDS

S. W. STRATTON, DIRECTOR

No. 118

A CRITICAL STUDY OF THE LEDEBUR METHOD FOR DETERMINING OXYGEN IN IRON AND STEEL

BY

J. R. CAIN, Chemist

and

EARL PETTIJOHN, Assistant Chemist

Bureau of Standards

ISSUED JANUARY 11, 1919

PRICE, 5 CENTS

Sold only by the Superintendent of Documents, Government Printing Office
Washington, D. C.

WASHINGTON
GOVERNMENT PRINTING OFFICE
1919

A CRITICAL STUDY OF THE LEDEBUR METHOD FOR DETERMINING OXYGEN IN IRON AND STEEL

By J. R. Cain and Earl Pettijohn

CONTENTS

	Page
I. Introduction	3
II. Apparatus and reagents	6
1. Hydrogen generators and purification of hydrogen	6
2. Furnaces, combustion tubes, and temperature control	10
(*a*) Furnace	10
(*b*) Boats	12
(*c*) Combustion tubes	12
(*d*) Temperature control	13
3. Weighed absorption tubes	14
III. Sources of error in operation of apparatus; blanks	14
1. Sources of error	14
(*a*) Removal of air	14
(*b*) Removal of moisture	15
(*c*) Time of heating	15
(*d*) Weighing phosphorus pentoxide tubes	15
2. Blanks	16
(*a*) Rate of passage of hydrogen	17
IV. Sources of error in preparation of samples	18
1. Sampling	18
2. Size of chips	18
3. Surface oxidation	19
4. Drying and preservation of chips	23
5. Discarding fine parts of samples	26
V. Reducibility of oxides apt to be present in steel	27
VI. Details of method used for determining oxygen	29
General conclusions	32
VII. Summary	33

I. INTRODUCTION

Oxygen in steel and iron is believed by most investigators to be present in the combined form. Indeed, it is difficult to conceive of elemental oxygen remaining as such in equilibrium with an excess of a solution of carbon in molten iron. The exact form of combination can only be conjectured. Metallographic methods for differentiating oxides in steels have not been developed, except

in a few special cases, and chemical methods dependent upon separating the oxides by selective solubility have proved unsatisfactory. Solutions of iodine, ferric chloride, cupric salts, and other solvents have often been proposed and tried for such separations, but the difference in solubility of metallic iron and of the oxides concerned has not been sharp enough to enable satisfactory results to be obtained.[1]

Oxides with known or unknown formulæ of the following elements may exist in steel: Class 1, iron and manganese; class 2, silicon (silicates), aluminum, chromium, vanadium, and titanium; class 3, copper, nickel, and tungsten; class 4, phosphorus and sulphur; class 5, carbon. Class 1 consists of metals present in quantity relatively large to that of the reducing agent (carbon). The corresponding oxides would, therefore, probably be present in steel. Oxides of metals in class 2 have very high heats of formation; hence are difficult to reduce by carbon except at temperatures higher than those usual in steel or iron furnaces, and are therefore probably present to some extent in the steels which contain these metals as essential ingredients. Oxides of metals in class 3 are, on the contrary, very readily reduced by carbon at temperatures prevailing in steel furnaces, and are probably never present in steel. Oxides of class 4 are volatile or decomposed at steel-melting temperatures and are not to be expected in the metal. Class 5 contains the oxides of carbon. These have been reported by numerous investigators, and it is almost certain that carbon monoxide, at least, is present.

Ideal methods for determining oxygen in steel should permit the separate determination of each form of combination. There is a theoretical possibility of accomplishing this, but there would be many practical difficulties. Some work of this kind has been done by the differential reduction of the oxides with various reducing agents and varying reduction temperatures, or by both. Thus, Pickard [2] has investigated in this connection the reducibility by hydrogen of manganous oxide, iron oxides, and oxide of carbon. Walker and Patrick [3] state that reduction by carbon in an Arsem vacuum furnace under the conditions prescribed by them gives the total oxygen content of steel, in contrast with the Ledebur method (reduction with hydrogen at 900 to 1000°), which, they claim, gives only the iron oxide content.

---

[1] Pickard; Carnegie Scholarship Memoirs, Iron and Steel Inst., 5, pp. 73–76.

[2] Cf. Carnegie Scholarship Memoirs, Iron and Steel Inst., 5, pp. 81–82.

[3] Proc. Eighth Internat. Cong. Applied Chem., 21, p. 139.

It seemed advisable to make a study of the Ledebur[4] method for determining oxygen in steel and iron, since this method has been used more extensively than any other proposed. The method as described by its originator, consists in heating drillings or chips of the metal in a current of pure hydrogen to about 1000°, collecting the water formed by reduction of the oxides and weighing it. The apparatus used by Ledebur consisted of generators for nitrogen and hydrogen followed by appropriate purifying apparatus for each. Generators and purifying apparatus were attached to one end of the combustion tube. To the other end of the combustion tube weighed phosphorus pentoxide tubes were attached, in which the water formed as a result of the reduction was collected. The procedure consisted in inserting the boat containing the chips into the cold combustion furnace, passing pure dry nitrogen over it to replace air, and heating the chips in nitrogen to remove moisture and organic matter. After cooling, the nitrogen was replaced by purified dried hydrogen, the temperature raised to the proper point, and the heating and passage of the hydrogen continued for 30 or 40 minutes. The phosphorus pentoxide tubes were then disconnected and weighed and the oxygen content of the steel calculated from their increase of weight.

Variations in the method introduced by others have been mainly in details, such as omission of the use of nitrogen,[5] weighing phosphorus pentoxide tubes filled with air instead of hydrogen,[6] electric instead of gas furnaces for heating,[7] improved forms of hydrogen generators, absorption tubes, etc. Very special changes in Ledebur's procedure and apparatus were introduced by Pickard,[8] who used a definite volume of hydrogen at less than atmospheric pressure in a confined space instead of passing a current of the gas through the combustion tube. He used a weighed boat as container for the phosphorus pentoxide.

In our study we have given special consideration to: (1) Apparatus, with a view to simplicity, reliability, small blanks in determinations, and durability; (2) sources of error; (3) preparation of samples for determinations; (4) reducibility, under the conditions of the Ledebur method, of various oxides likely to be present in steel; and (5) accurate determination, by the Ledebur method, of oxygen in some typical American irons and steels.

---

[4] Leitfaden für Eisenhütten Laboratorien, 9th ed., p. 114.
[5] Cushman, J. Ind. and Eng. Chem., 3, p. 372.
[6] Ibid.
[7] McMillen, J. Ind. and Eng. Chem., 5, pp. 123–125.
[8] Carnegie Scholarship Memoirs, Iron and Steel Inst., 5, p. 77.

II. APPARATUS AND REAGENTS

1. HYDROGEN GENERATORS AND PURIFICATION OF HYDROGEN

One of the hydrogen generators used for our work is shown in Fig. 1. It is similar in design to generators that have for some time been successfully used in the section of gas chemistry at this Bureau. To operate the generator, fill the bottle *F* with zinc (see Fig. 1) and replace the stopper, closing the bottle. Fill the reservoir *B* with dilute sulphuric-acid mixture 1 to 6, containing 5 g.$FeSO_4.7H_2O$ per 100 cc. Open the cocks *C* and *H*, which permit hydrogen to escape (usually through the train) and apply air pressure to the acid reservoir, *B*. Acid is thus driven over into

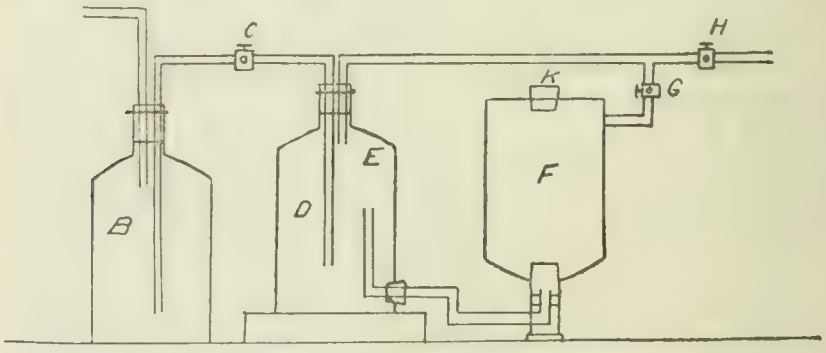


FIG. 1.—*Hydrogen generator*

D. When sufficient acid has been forced into *D*, stopcock *C* is closed and the air pressure released. A constant, regulated current of hydrogen is then obtained by manipulating cock *G* (*H* being, of course, open). To stop the generation of gas, close the cocks *G* and *H* and open cock *C*. The pressure of the hydrogen being generated in *F* forces the acid out of *F* and *D*, partially. As soon as the acid is all driven away from the zinc, gas ceases to be generated.

This generator has been found very convenient in use and has advantages over others that have been proposed for this kind of service. After being filled with reagents and freed from air (by displacement or evacuation, or both) spent acid can be replaced without any air being introduced into *D*. *F* holds enough zinc to suffice for about 30 determinations. The flow of hydrogen can be well regulated without much variation of pressure inside the apparatus. The position of the acid inlet at *E* insures fresh acid being in contact with the zinc, the spent acid, because of its

greater density, settling to the bottom of *D*. Because the generator is left under atmospheric pressure there is minimum danger from overflow or loss of acid or from blowing out the rubber stoppers in the generator or apparatus.

The impurities to be removed from hydrogen generated in this manner are: Water vapor, oxygen (from air), carbon dioxide, carbon monoxide, and some hydrogen sulphide. The possibility of carbon monoxide being present seems to have been overlooked by others, since we find no reference to it in the literature. Its absence in the gas entering the combustion tube by way of *G* (Fig. 3) is desirable, since hydrogen slowly reduces carbon monoxide (see pp. 28 and 29) under the conditions of the Ledebur method, and the water vapor resulting from this would give high results for oxygen in steel samples. It is less desirable to correct this error by a blank than it is to eliminate the carbon monoxide altogether; hence we have adopted the latter method. Both the carbon dioxide and monoxide come from the zinc, and it is interesting to note that samples tested from three different sources all contained both gases, and only one, a zinc from an English source unknown to us, was free from this gas. Since we happened to have an abundant supply of this zinc, it was used in all of our work with this generator. Consequently, the apparatus shown in Fig. 1 does not provide for the removal of carbon monoxide. Probably the simplest method for accomplishing this would be to pass the hydrogen through a U tube (both limbs sealed at the top, no rubber stoppers or lubricated stopcocks being permissible) filled with iodine pentoxide and heated by an oil bath to 150°. This would oxidize the carbon monoxide to dioxide, which would be absorbed by soda lime. Hydrogen would not be oxidized under such conditions. Since, as stated, we used carbon-monoxide-free zinc, no iodine pentoxide tube such as proposed is shown preceding the tube *G* (Fig. 3), whose function is to remove the carbon dioxide always present in the hydrogen. If a carbon-monoxide purifier of the type described is used, it should be inserted between the hydrogen generator and *G* (Fig. 3). The soda-lime tube *G* also serves to remove any hydrogen sulphide. The removal of the traces of oxygen always likely to be present from air, even when the greatest care is used in freeing the generator of air, is secured by the catalyzer. This consists of a ¼-inch bore tube of porcelain, glazed in this case inside and out, heated to 600° for 6 inches in the middle by the electric furnace *F* (Fig. 3). This tube is

filled loosely in the heated part with platinized quartz. The phosphorus pentoxide tube, *D*, removes the water vapor formed

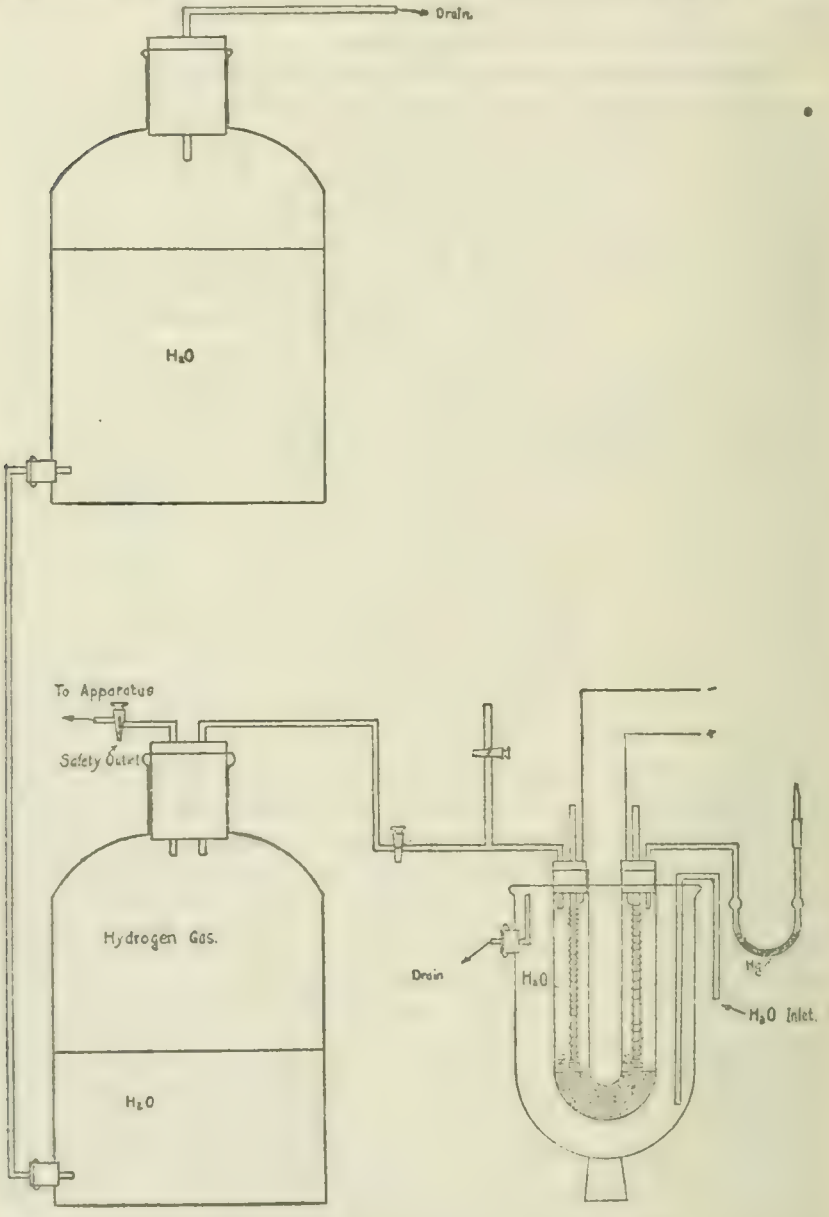


FIG. 2.—*Electrolytic hydrogen generator*

by combustion of the oxygen present as impurity, as well as the water vapor originally present in the gas.

Electrolytic Hydrogen Generator and Reservoir.—Hydrogen gas was generated by the electrolysis of a saturated solution of barium-hydroxide mixed with a 25 per cent solution of sodium hydroxide in a large pyrex glass U tube, using a platinum anode and a nickel cathode. Platinum and nickel were used as the electrode materials because they have a low oxygen and a low hydrogen overvoltage, respectively. The generator (Fig. 2) consists of a U tube containing the electrolyte and submersed in a jar through which flows cold water. The bottom of the U is filled with sea sand to hinder the passage of dissolved gas from one limb of the U to the other, as suggested by Lewis, Brighton, and Sebastian.[9] The current passing through the generator is regulated by means of a rheostat in the circuit. During the course of the investigation the current used by the operator was 3.3 amperes, which liberates about 1.5 liters of hydrogen gas per hour.

The hydrogen gas reservoir and pressure-maintaining bottle is connected to the cathode side of the generator, and to the anode side a small U tube of 10 mm inside diameter is attached which contains mercury to balance the pressure in the receiving system. With this arrangement the generator operates automatically. As hydrogen is generated and delivered to the gas reservoir water is displaced from the gas reservoir and forced into the water bottle above. The increased pressure thus produced forces down the liquid in the cathode side of the generator an amount approximately equal to the height to which the water level in the water bottle is raised. After a certain volume of hydrogen gas has been generated and stored in the reservoir, the level of the electrolyte on the hydrogen side of the generator will be forced down out of contact with the cathode, thus automatically breaking the circuit. The pressure of the hydrogen that is desired for the experiment is regulated by adjusting the height of the water bottle, which then determines the amount of mercury that must be added to the U tube on the oxygen side of the generator. The hydrogen gas as it is drawn off from the reservoir for use is passed through the catalyzer and purifying train.

The electrolytic hydrogen generator is followed by the same type of purifying train as shown for the zinc generator. No carbon monoxide need be feared in using this method of generating hydrogen.

[9] J. A. C. S., 39, 1917, 2248.

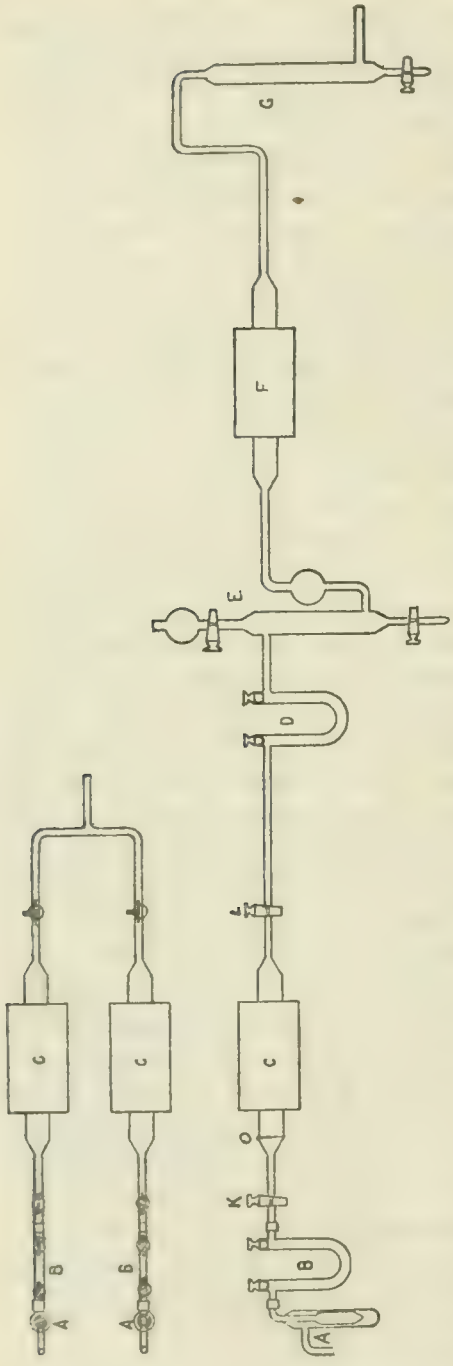


FIG. 3.—*Apparatus used for determination of oxygen*

2. FURNACES, COMBUSTION TUBES, AND TEMPERATURE CONTROL

(*a*) *Furnace.*—An electric furnace is very greatly to be preferred for Ledebur oxygen determinations. Gas furnaces may be and have been used, but they are much harder on tubes, and in case of porosity or minute cracks in the latter the danger from inwardly diffusing water vapor from products of combustion is very great, a difficulty which is entirely obviated by electric heating, which we have used in our work. The type of furnace shown at *c* in Fig. 3 and in detail in Fig. 4 was used. Obviously, in work of this kind another combustion can not be begun until the entire apparatus has cooled practically to room temperature after the preceding combustion. But since, in general, for efficiency in heating the thermal insulation of an electric furnace should be made very effective, it follows that cooling is correspondingly slow. The difficulty of a slow-cooling furnace can be obviated by (*a*) fitting the furnace with a removable heat-insulating jacket; (*b*) by using a combustion tube closed at one end, thereby dispensing with a rubber stopper and connections at that end, so that the hot furnace may be

slid on and off there; (*c*) by providing heat insulation incased in a sheet-iron jacket split and hinged in the medial horizontal plane, so that the hinged halves may be opened and the furnace removed from the tube without interrupting the heating current. Method (*c*) was considered the most convenient, and the hinged furnace shown in Fig. 4 was built according to our specifications for this work.[10] This furnace is satisfactory and by its use with a porcelain tube a new determination may be begun

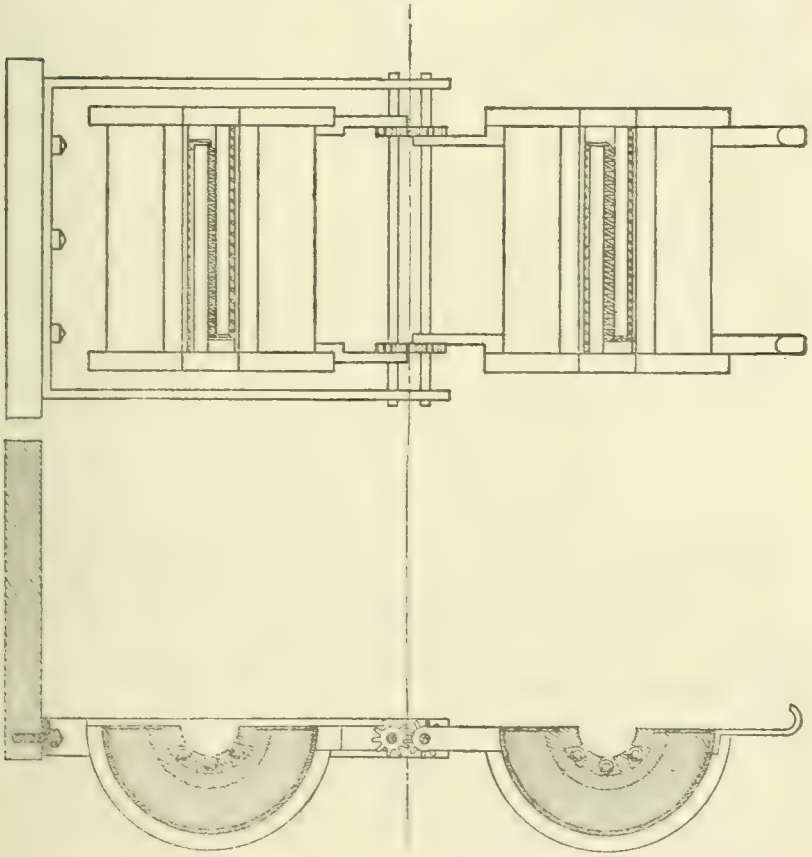


FIG. 4.—*Special electric furnace for oxygen determinations*

within 20 minutes after completing the preceding one. With most porcelain tubes the opening of the furnace and the closing of the hot sections upon the cold tube for heating must be done rather gradually, owing to the danger of rupturing the tube by sudden temperature change. This is particularly true with the somewhat inferior porcelain now available. If fused silica tubes are used,

[10] A great deal of work was also done in the ordinary nichrome-wound electric furnaces without removable heat insulation such as are in general use at the Bureau.

this condition is much improved. However, as will be seen later, the fused silica tubes which we have tried for the work are open to certain objections.

(*b*) *Boats.*—Boats may be made of quartz, porcelain, nickel, or iron, the order of preference being as given. We find statements in the literature on the Ledebur method to the effect that quartz reacts with iron or hydrogen at the working temperatures of the Ledebur method, but these statements are not confirmed by our experience. Porcelain boats are less desirable than quartz, owing to the greater liability to fracture by sudden temperature changes. Nickel and iron boats, the latter in much greater degree than the former, are liable to oxidation if removed from the furnace when they are at all hot. In fact, even at ordinary temperatures there is some oxidation of both metals on long standing, so that they must be prepared for use by preliminary reduction in hydrogen if the operator is to feel safe on this point. The size of the boat is governed by the physical form and the amount of chips used for each determination. We have found 20 to 30 g of chips a suitable weight, and if the size of chip recommended later (p. 19) is used, a boat of about 15 cc capacity is required. Our quartz boats measured 15.0 cm by 1.3 cm by 1.3 cm inside. The bottoms were flat and slightly concave inside toward the edges.

(*c*) *Combustion tubes.*—The use of proper combustion tubes is imperative if reliable results are to be expected. The desirable qualities for work of this kind are: Freedom from tendency to oxidize, mechanical strength, impermeability to oxygen or to water vapor, inertness toward hydrogen at 900 to 1000°, resistance to temperature changes, and absence of structural changes by heat, with consequent development of porosity. Platinum tubes are perhaps best, but are expensive. Fused silica tubes are good until they become opaque, due to the gradual formation of tridymite, when they develop porosity. (See Table 1 (1).) The useful life of quartz tubes is, on this account, rather short. Opaque or semitransparent quartz tubes, so far as our results show, are very dangerous unless they are thoroughly glazed. Blanks to ascertain the condition of tubes should be run every few days, as deterioration is sometimes quite rapid. When the blank per one-hour heating exceeded 0.0003 g, we put in new tubes for our most accurate work [11] unless the blank was found to be due to other causes. Porcelain tubes now available are of variable quality. Koyal Berlin tubes, some with inside glaze only, others with both inside and

[11] See footnote under Table 1.

outside glaze, were used, and, in general, were found quite satisfactory. Occasionally defective porcelain tubes from the most reliable sources are found (Table 1 (2, *a*).) The only safe way is to test each tube thoroughly before installing and to run frequent blanks afterwards until failure is detected. In more recent work Copenhagen porcelain tubes and Imperial porcelain tubes have been used with fair satisfaction. No American-made tubes yet submitted to us have proved at all reliable. The outstanding objection to porcelain is the poor resistance to sudden temperature changes, an objection which is probably inherent and which sets the limit on the time required for an individual determination.

TABLE 1.—Data on Combustion Tubes

Kind	Dimensions	Blank (boat present)	Time of each heating	Blank when discarded [a]	Life hours, at 1000°
	cm	g	hours	g	
(1) Quartz	60 by 2.2	0.0010	1	0.0088	250
		.0014	2		
		.0017	3		
(2) Porcelain:					
(*a*) Royal Berlin	60 by 2.2	0.0020 0.0024	4	.0045	150-200
	60 by 2.2	.0010	1		
		.0040	4	Not used	
(*b*) Royal Imperial	60 by 2.2	.0010	1	.0040	120
					[b] 70
					[b] 166
(*c*) Royal Copenhagen	60 by 2.2	.0010	1	.0050	66
(*d*) Fused silica (unglazed	60 by 2.5	.0036- .0044	4	Not used	

[a] The blank denotes increase in weight of the phosphorus pentoxide tube. The increase in blank noted when the tube is discarded occurs all at once and is not a gradual increase from 0.0010 g per hour to the figure given in this column. The normal blanks indicated here were obtained during the first stages of our work, and were later reduced to 0.0003 g per hour. The values are given because they show the quality of the varieties of tubes used. See Blanks, p. 17.

[b] Still in use.

(*d*) *Temperature Control.*—The usual working temperature in Ledebur determinations is 900 to 1000°, at which temperatures the oxides of classes 1, 3, and 5 (see p. 4) are reduced wholly or partially.[12] No advantage is to be gained by carrying the working temperature to the maximum practicable when working with glazed porcelain tubes (1200 to 1400°), since these temperatures are still below the temperature range where the lower oxides of class 2 are reduced to metal by hydrogen but are above the temperature range where porcelain tubes have a long life. The most convenient method for measuring the working temperature is by base metal or by platinum-rhodin thermocouples connected to

[12] Some work showed that a very considerable reduction takes place at temperatures as low as 500 or 600°.

direct-reading millivoltmeters. In our work both types of couple were used. The temperature of the furnace should be measured every three or four days of actual service. A steady temperature drop not caused by change in voltage of the current supplied the furnace is probably due either to oxidized terminal connections or to deterioration of the resistor, and should immediately be remedied.

3. WEIGHED ABSORPTION TUBES

Glass-stoppered U tubes of the type usually employed for carbon determinations were used and found satisfactory. The limbs of our tubes were 12.5 cm long and 0.90 cm in bore. They were fitted with well-ground stoppers kept well lubricated with a good quality of stopcock grease. They were filled with alternate layers of phosphorus pentoxide and glass wool in such manner that free passage of gas was not impeded. Counterpoises of the same approximate shape and size were used in weighing. A guard tube *A* (Fig. 3) containing concentrated sulphuric acid was used at the end of the train to prevent access of moisture from the air to the weighed tube.

III. SOURCES OF ERROR IN OPERATION OF APPARATUS AND BLANKS

1. SOURCES OF ERROR

The principal sources of error in Ledebur oxygen determinations other than those already described and the errors resulting from faulty preparation of the sample (see next section) are: (1) Impurities in the hydrogen; (2) too low temperature; (3) failure completely to remove air from the combustion tube after inserting the boat containing the sample, (4) moisture condensed on the sample; (5) too short a period of heating during the determination; (6) weighing phosphorus pentoxide tubes; (7) rate of passage of hydrogen.

Errors (1) and (2) have been dealt with in Section II.

(*a*) *Removal of Air.*—Completeness of removal of air from the apparatus was tested by taking successive samples of the gas from the exit end of the combustion tube in a test tube during rapid passage of the hydrogen until a sample no longer exploded, tests being made every two minutes.[13] An oxygen content of less than 4 per cent was thus indicated, and passage for 10 minutes longer

[13] Some experiments were made in which air was removed by evacuation with a good water aspirator, refilling with hydrogen and again evacuating. This method was very effective.

assured an error of less than 0.001 per cent oxygen on a determination from this cause, assuming a weight of 30 g of steel to be used. This value has been checked by numerous blank determinations, detailed later.

(*b*) *Removal of Moisture.*—Since the incoming hydrogen is dried over phosphorus pentoxide, the simultaneous removal of any moisture in the air being displaced, or of any condensed moisture on the sample is assured. Our method of preparing samples also practically excluded the possibility of surface moisture.

(*c*) *Time of Heating.*—(See Table 2.) By varying the time of heating of several samples of steel taken from the same bar (Bureau of Standards sample No. 23, Bessemer, 0.8 per cent carbon) from one to four hours at 1000°, we found a one-hour period sufficed for complete removal of all oxygen determinable by the Ledebur method. This was checked on several samples. After the one-hour heating period the sample was heated without removal from the furnace for an additional hour in a great many cases and showed a negligible amount of oxygen, i. e., less than 0.001 per cent on 30 g from the second hour's heating.

TABLE 2.—Oxygen Results when Amount of Sample and Time of Heating are Varied

Sample	Time of heating	Weight	Oxygen
	Hours	g	Per cent
Source unknown	1	15	0.041
	1	20	.041
B. O. H., 0.6 per cent carbon	1	30	.013
		25	.012
B. O. H., 0.2 per cent carbon	1	30	.015
		15	.016
		15	.015
Ingot iron	1	15	.036
		20	.035
		30	.035
Chrome vanadium steel	1	30	.009
		25	.009
Bessemer, 0.8 per cent carbon	1½	30	.011
	1	30	.011
	4	30	.010
	4	30	.012
	4	30	.010
	1	30	.011
	1	50	.012

(*d*) *Weighing Phosphorus Pentoxide Tubes.*—Two methods have usually been employed by others for weighing phosphorus pentoxide tubes: (*a*) Filled with air by an aspirator connected with

phosphorus pentoxide drying tubes; (*b*) filled with hydrogen, i. e., as they are when disconnected from the combustion apparatus. Objections have been raised to the latter method because of the fear that hydrogen would be too readily lost by diffusion through stopcocks or stoppers. We have had no difficulty of this kind when using the tubes and stopcock lubricants previously described, and hence have used this method for all our work. Tubes were allowed to stand a half hour near the balance before final weighing. For comparison the initial weight just after disconnecting from the combustion tube was also recorded. The weight the following morning and the weight after a half hour differed usually by not more than 0.3 mg, a change which may be largely attributed to external atmospheric causes, since tubes filled with air will show under the same circumstances almost the same variation. This slight change in weight after 12 hours' standing shows how effectively the hydrogen is held within the tube. Table 3 shows further experimental data on this point. As previously stated, counterpoises of the same approximate weight and size as the tubes themselves were used in weighing.

TABLE 3.—Successive Weights of Absorption Tubes Filled with Hydrogen

Time	Weight of tube 3 [1]	Weight of tube 24
	g	g
Gas passed 2 hours	2.0224	0.2711
Stood ½ hour	2.0221	.2706
Stood 48 hours	2.0217	.2701
Stood 72 hours	2.0214	.2697
Stood 96 hours	2.0212	.2695
Stood 120 hours	2.0215	.2697
Stood 144 hours	2.0212	.2696
Stood 168 hours	2.0212	.2694
Increase in weight in 168 hours	.0012	.0017

[1] The columns give the weights that were placed on the pan with the absorption tube to balance the counterpoises used.

2. BLANKS

Blanks were run with the empty boat inserted, the furnace and catalyzer being kept at the usual working temperatures. Before heating the test for complete removal of oxygen was made as already described.

Under these conditions a blank of from 0.0010 to 0.0015 g of water has been found by those who have made use of this method.

Our early work, including that done on testing the various grades of porcelain tubes (Table 1) showed the same blank.

It was found that if the preheater was kept hot for an hour with the gas flowing through the train before the furnace tube was heated, the blank dropped to 0.0006 g of water. On replacing the rubber stoppers originally used to close the open end of the combustion tube, with glass caps sealed on with Khotinsky wax, the blank again dropped, the increase in weight for an hour at red heat being from 0.0001 to 0.0002 g. Since this represented less than 0.001 per cent oxygen on a 30 g sample, it was considered satisfactory.

The average frequency of our blank determinations was about 1 per 6 or 7 determinations unless there was reason to suspect the presence of defects in tubes, boats, or other parts of the apparatus. As soon as a blank greater than 0.0003 g was obtained in the later work and was found to be irremediable, the tube giving it was replaced by a new one.

Some of the blanks obtained by the original method and by the method as finally used are shown in Table 4.

TABLE 4.—Blanks on Furnace Tube with Empty Boat Present. Period of Blank 1 Hour [a]

1. Original method	2. Preheater hot for 1 hour	3. Preheater hot for 1 hour; glass caps
0.0012	0.0004	No blank
.0011	.0005	No blank
.0010	.0006	0 0001
.0008	.0007	.0002
.0010	.0005	.0001
.0008	.0006	.0001
.0010	.0006	

[a] The values are expressed in grams of water obtained.

(*a*) *Rate of Passage of Hydrogen.*—Work done at the Bureau on the equilibria between hydrogen, carbon, and oxide of iron referred to later in this paper (p. 28), shows that unless a rate of passage of hydrogen not less than 60 cc per minute is maintained part of the oxide is reduced by the carbon with formation of CO or CO_2, and consequently an equivalent amount of oxygen is lost in the Ledebur method, which, of course, measures only the oxygen liberated as H_2O. With very slow rates of hydrogen passage the error may be very serious.

IV. SOURCES OF ERROR IN PREPARATION OF SAMPLES

By far the greatest possibilities for error in Ledebur determinations lie in the preparation of the sample. Unless necessary precautions are taken in this regard, the results are liable to be worthless. (See Tables 5 and 6.) Errors may be due: (*a*) To improper choice of places for taking the sample, such as localized rust or oxide spots, slag streaks, and oxidized blowholes, or to improper sampling of the mass of metal itself; (*b*) too large chips; (*c*) surface oxidation during cutting of chips, because of too high tool speed, hard metal, dull tool, etc.; (*d*) discarding fine parts of the sample, as often recommended, because of the fear that the fine parts are most liable to be surface-oxidized during cutting; (*e*) moisture condensed on the sample, or contamination by other substances containing oxygen, e. g., scale.

1. SAMPLING

In our opinion not enough work has been done on the question of oxygen distribution in steel to enable any rules for sampling to be formulated. The only recommendation that can be made at present is to follow the procedure used in sampling when other constituents are to be determined and to avoid the localized oxygen zones just mentioned, unless definite information regarding these is sought. Two of our samples (standard sample 8a and an ingot iron) have been analyzed for oxygen a number of times, so that the results show oxygen values over a considerable volume of the original material. These results check to 0.002 per cent. On duplicate determinations as usually made we have not found the oxygen content to vary more than the error of the determination (0.002 per cent oxygen) throughout volumes of 16 to 20 cc of the various (forged or rolled) steels mentioned in Table 9.

2. SIZE OF CHIPS

We have conducted several experiments to determine the thickest chip permissible. Pickard [14] concluded from his work that chips should not exceed 0.2 mm in thickness. However, Pickard's samples were cut in air, and there is therefore some slight uncertainty on this account, since he gives no indication of the probable error from surface oxidation. Table 5 shows the results obtained by us with various thicknesses of chips from the same sample, all cut under oil, as described subsequently. From this we conclude

[14] Carnegie Scholarship Memoirs, 5, pp. 81-84

that chips should not be thicker than 0.15 mm. Any advantage gained by using a thinner chip (such as possible shortening of time required for a determination) is likely to be offset by the longer time required in the shop to prepare a sample.

TABLE 5.—Variation in Apparent Oxygen Content with Thickness of Chips

Sample	Thickness of chip	Oxygen	Result by—
		Per cent	
Bessemer, 0.4 per cent carbon (No. 10B)	0.015 cm	0.014	Authors
	Coarse from same	.007	Do.
	Fine from same	.019	Do.
A. O. H., 0.4 per cent carbon (No. 20A)	0.015 cm	.023	Do.
	Coarse from same	.020	Do.
	Fine from same	.029	Do.
Unknown	0.015 cm	.034	Do.
	Coarse from same	.024	Do.
	Fine from same	.035	Do.
Ingot iron	0.005 cm	.036	Do.
Do	0.015 cm	.035	Do.
Do	0.030 cm	.033	Do.
Do	0.045 cm	.031	Do.
Do	0.055 cm	.030	Do.
Do	0.075 cm	.027	Do.
Do	0.100 cm	.017	Do.
Basic open-hearth, billet I	0.010 cm	.019	Pickard
		.020	Do.
	0.015 cm	.019	Do.
		.017	Do.
	0.070 cm	.013	Do.
		.008	Do.
	Fine material	.020	Do.
Basic open-hearth, billet II	Average	.014	Do.
	Finest	.027	Do.
	Thickest	.002	

3. SURFACE OXIDATION

We recognized from the first that the surface oxidation of samples during cutting might be very serious. This point has been mentioned many times by others, but the methods given for obviating the difficulty have seemed to us either inadequate or the descriptions of these methods lacking in necessary detail. It has just been shown by the results of Table 5 that a very thin chip is necessary. This causes the surface of the cut sample to be very large. The work done in cutting it, and consequently the heat generated during cutting, is very great compared with the surface exposed and heat generated when cutting the larger-grain samples recommended by some authors. Consequently the liability to surface oxidation was greater with us than with those who used

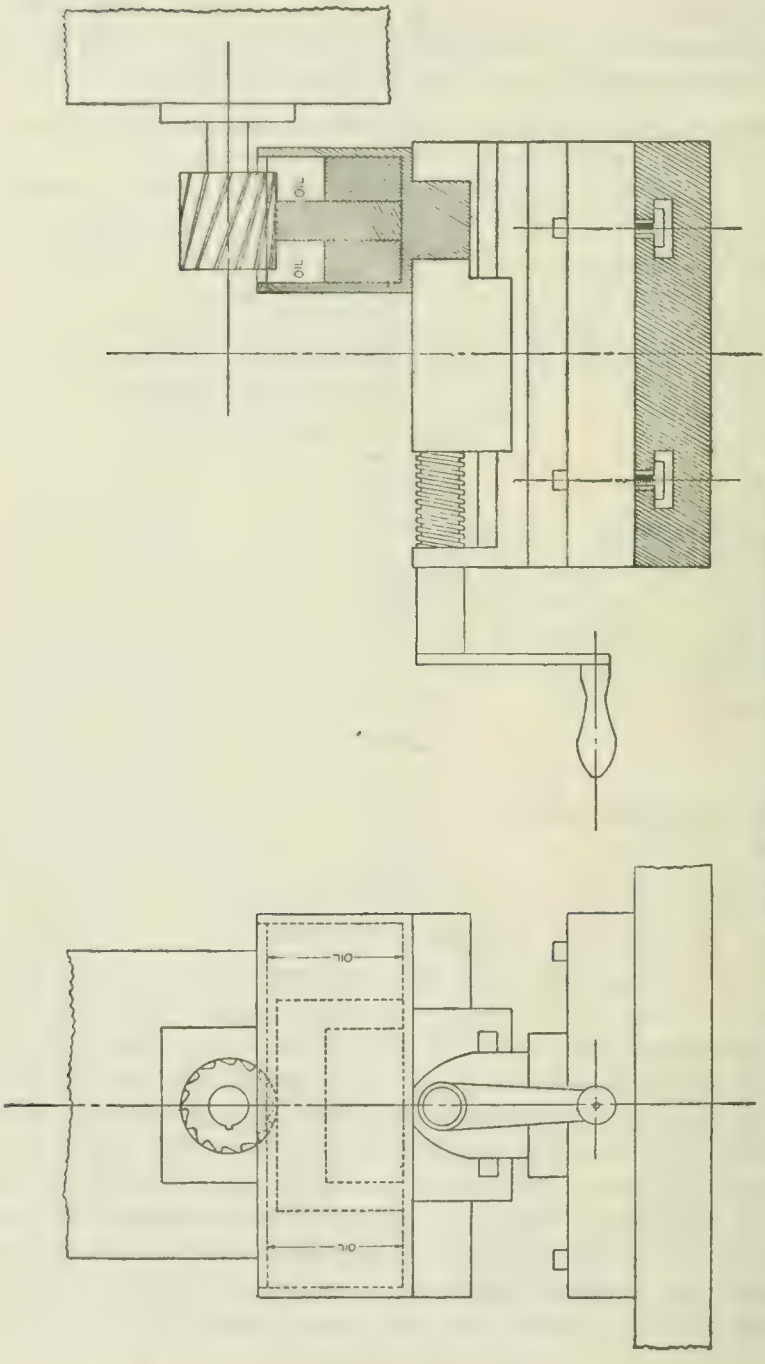


FIG. 5.—*Apparatus for cutting samples under oil*

larger chips. To avoid errors from this cause we tried various methods of cutting the samples under nonoxidizing, noncorroding liquids, in order to exclude air and at the same time to reduce local overheating at the point of contact of the tool with the work through the cooling effect of the large mass of liquid under which cutting was done. Mercury seemed at first to be ideal for this purpose, but practical difficulties were encountered in its use, because of its peculiar tendency to become coated, after the cutting operation has continued for a minute or so, with a scum which gradually increases in amount and from which the chips can not be freed. Even the purest mercury at our disposal showed this peculiar phenomenon. Another difficulty was the tendency of the chips to float to the surface of the mercury where they are liable to oxidation. Pure benzene was substituted as protecting liquid, but although successful, was too volatile to be convenient. Of course, the portion volatilized was lost beyond recovery and the method was therefore expensive. Pure transformer oil (H. F. grade, Westinghouse Electric & Manufacturing Co.) was next tried, and found entirely satisfactory after a suitable procedure for washing the chips free from it was developed. Fig. 5 shows the device used for cutting under oil. The chips were taken on an ordinary milling machine, a metal box being clamped between the jaws of the vise. After cutting a sufficient amount of sample from the piece of metal (which had previously been freed from surface oxide, dirt, etc.), the box is removed by opening the jaws of the vise on the milling machine, the excess of oil drained off into a suitable container, the sample poured into a bottle and kept covered with the transformer oil until ready to be washed free from oil. Very fine particles of iron tend to remain suspended in the used oil, and, because of their minute state of division, they will oxidize after the oil has stood for some time; hence it is not advisable to use the oil repeatedly unless it is filtered through a sand column or otherwise freed from this contamination.

The chips are washed free from oil by transferring them to a glass-stoppered bottle, and covering them with benzene. After shaking, the benzene is decanted off and a fresh portion added. After six washings, the benzene shows no discoloration from oil. The chips are then thrown on a Büchner funnel and washed three times with pure benzene, suction being applied, and the excess liquid removed, after each addition.

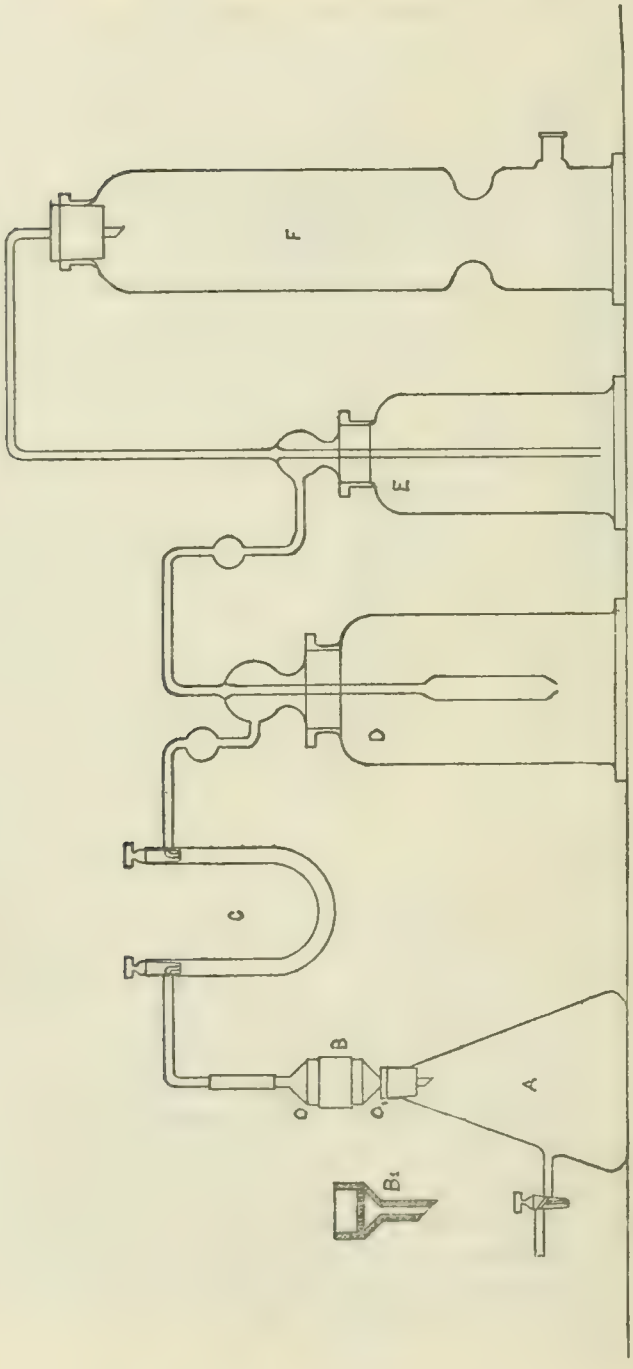


FIG. 6.—*Apparatus for drying samples*

4. DRYING AND PRESERVATION OF CHIPS

The final operation of preparing a sample for analysis is carried out in the apparatus shown in Fig. 6, and consists in fitting cover O to O_1 and drying the sample by sucking dry air through it. The air passed through a plug of cotton, F, then successively through alkaline permanganate, E, concentrated sulphuric acid, D, phosphorus pentoxide, C (right limb), and a short column of reduced steel, C (left limb). Fig. 7 shows another form of apparatus which was used for drying many of our samples after they had been washed as described. The used benzene can, of course, be recovered by distillation.

FIG. 7. *Drying tube*

Experiments with ether and gasolene as washing agents were unsuccesful, owing to large blanks introduced into the analysis by their use. These may be due to the retention on the chips of nonvolatile organic impurities containing oxygen. It is absolutely necessary that dry air be used in aspirating, otherwise the cooling of the sample below room temperature by the rapid evaporation of the benzol causes the condensation on the sample of atmospheric moisture.

After the samples have been washed and dried as described, an operation that should not be delayed for more than a few hours after cutting, they are put in glass containers and kept in a desiccator over sulphuric acid or phosphorus pentoxide until oxygen is determined in them.

Table 6 giving comparative results on the same samples milled in air and under oil, respectively, shows the necessity of cutting under oil to insure freedom from errors arising from surface oxidation. Such errors are variable and may, as shown in the table, range from 15 per cent to 170 per cent of the oxygen present.

TABLE 6.—Comparative Values for same Samples Milled in Air and Under Oil

Sample	Under oil, in per cent oxygen [a]	In air, cut at low speed, in per cent oxygen [a]	Increase in air, per cent
B. O. H., 0.2 carbon	0.014	0.021	50
A. O. H., 0.2 carbon	.023	.034	48
Bessemer, 0.4 carbon	.014	.022	57
B. O. H., 0.4 carbon	.012	.017	42
A. O. H., 0.8 carbon	.010	.027	170
Ingot iron	.035	.042	20
Bessemer, 0.1 carbon	.020	.023	15

[a] The chips cut in the air and under oil were of the same size.

We have found a uniform blank due to preparation of sample and equivalent to 1.2 mg of oxygen per 30 g sample on all chips cut under oil as described. This is equivalent to 0.004 per cent oxygen. The size of the blank was determined by subjecting a sample of steel previously reduced in hydrogen to the same treatment employed in washing and drying the ordinary samples. An investigation of the source of this blank and of the possibility of eliminating it was also made.

There were a number of factors that might account, wholly or in part, for the observed blank. It might be due to impurities in the oil or in the benzene. An oxidation of the sample might take place inside the furnace, either through leakage of oxygen when rubber stoppers were used, or through oxygen passing the preheater while it is cooling and then uniting with the cooling, reduced iron in the furnace. Dry air might also form an oxide film on the surface of the reduced steel during the drying.

To investigate the source of the blank, parallel determinations on the same reduced samples were carried out, oil being used in the first, followed by benzene washing and air drying, and only the benzene washing and air drying in the second. The same blank was obtained in both cases for several different samples, which showed that the oil was not the source of the blank. (Table 7 (1).)

When glass caps were substituted for rubber stoppers at the end of the combustion tube, it was possible to reduce a sample, cool it in the furnace, and repeat the reduction the next day without danger of exposing to air. A sample so reduced gave a blank of less than 0.0003 g of water or less than 0.001 per cent on a 30 g sample. Moreover, this completely reduced sample showed the same blank on reduction after again washing it with benzene and drying it.

In view of these results it was concluded that the blank must be due either to impurities in the benzene or to oxidation by dry air. Parallel blank determinations were again carried out, (1) washing a reduced steel sample with benzene and drying as usual, and (2) passing dry air over another portion of the reduced sample for the same length of time as was required to dry a washed sample. Blanks obtained by reducing samples treated in these two ways again were of the same magnitude (Table 7 (2)). This led to the belief that the reduced steel took up oxygen from dry air to the extent of 0.004 per cent.

To test this, dry hydrogen was substituted for dry air to carry away the benzene and dry the sample. The results were very slightly lower, but still gave 0.003 per cent or 0.004 per cent, showing that the use of hydrogen did not eliminate the blank,

probably because the sample was exposed to the air in transferring from the drying apparatus to the furnace.

Finally, the completely reduced sample was exposed to air for a short time in the cold furnace. This was done by evacuating the furnace somewhat and allowing air to flow in and take the place of the hydrogen removed. The air was then removed, hydrogen admitted, and the same subsequent heating procedure followed as was used when the empty furnace gave a negligible blank. Samples so treated and kept in contact with air for only five minutes gave a blank of 0.003 per cent (Table 7).

Variation in the weight of the reduced sample used changed the weight of water obtained correspondingly, so that the oxidation of the sample was proportional to its weight, and the percentage of oxygen obtained was the same whether 10 g were used or 30 g.

Large samplies were sifted, so as to remove the finest chips present. A 60-mesh copper sieve was used for this purpose. Fine and coarse material from the same sample was reduced, the fine portion giving the larger amount of oxygen. The reduced sample was removed from the furnace, crushed in a clean agate mortar (since it sinters together in the furnace), and then returned to the furnace. On repeating the reduction, the fine material again showed a larger amount of oxygen. The samples used show the oxygen content of the coarse material to be about half of that of the fine (Table 7 (5)). This verifies the assumption we made when the oxygen was found to vary with the weight of sample taken, and proves that the oxidation is a surface phenomenon.

TABLE 7.—Blanks Obtained on Washing Reduced Samples with Oil and Drying

(1) EFFECT OF USE OF OIL

Blank (oil used), in per cent oxygen	Blank (no oil used), in per cent oxygen
0.004	0.004
.005	.005
.005	.005

(2) EFFECT OF USE OF BENZENE

Benzene washing and air drying, in per cent oxygen	Air drying for same time, in per cent oxygen
0.004	0.004
.005	.005
.004	.006
.004	.005
.005	.004
.003	
.005	

(3) EFFECT OF DRY AIR AND OF DRY HYDROGEN

Dry air, in per cent oxygen	Dry hydrogen, in per cent oxygen
0.004	0.004
.005	.0035
.006	.003
.005	
.004	

(4) EFFECT OF EXPOSING TO AIR IN THE COLD FURNACE

Per cent oxygen
0.0027
.0029
.0030

(5) OXIDATION OF COARSE AND FINE REDUCED MATERIAL FROM THE SAME SAMPLE

Sample	Coarse portion, in per cent oxygen	Fine portion, in per cent oxygen
Electrolytic iron	0.002	0.0055
Steel	.003	.008
Ingot iron	.004	.008

The conclusion reached as a result of this work on the blank is, therefore, that steel chips as we prepare them for Ledebur oxygen determinations are oxidized slightly and almost immediately on contact with air after the benzene is removed, the amount of the oxidation for the materials we used being about 0.004 per cent oxygen. The experimental work done also shows that it is impossible to avoid this oxidation unless a method is employed which entirely prevents exposure to the air from the time that the sample is milled. Such a method seems too difficult to have any practical value. It is also evident that samples cut in contact with air, as has been the usual practice heretofore, must be subject to a very much greater oxidation than by our method, which reduces this error to the practicable minimum. Such conclusion is amply confirmed by the results in Table 6. The speed of cutting these samples in air was probably lower than has been commonly used.

5. DISCARDING FINE PARTS OF SAMPLES

We regard this practice, so universally recommended, as inadmissible, because of possible segregation of oxides in the fine parts of the sample in certain cases. This is balanced to an extent by

the greater air oxidation of very fine particles during preparation of the sample (see Table 8), but such error is probably less than would be caused in many cases by rejection of the fine part of the sample. All of our work on coarse and fine samples (see preceding section) supports the assumptions made above and leads to the conclusion that in general the finer chips of the milled samples are higher in oxygen than the coarser ones. See also results on Johnson pig iron, in Table 10.

V. REDUCIBILITY OF OXIDES LIABLE TO BE IN STEEL

This subject has already been referred to in the introduction to this paper. Experimental work has been carried out by Pickard,[15] and Table 8 gives the results obtained by Pickard and ourselves.

TABLE 8.—Reducibility of Oxides Liable to be Present in Steel

Oxide reduced	Water calculated for complete reduction	Water found	Indicated reduction to—	Time of heating, in hours	Determination by—
Magnetite ore [a]	0.0135	0.0135	Fe	4	Authors
	.0177	.0174			
Fe_2O_3, pure	.0284	.0282	Fe	.....	Pickard
	.0245	.0241			
Manganese ore [b]	.0186	.0097	MnO	4	Authors
	.0076	.0038			
	.0610	.0298			
	.0380	.0208			
	.0512	.0278			
Manganese ore and magnetite ore	.0169	.0086	MnO and Fe	6	Do
	.0312	.0312			
	.0114	.0055			
	.0521	.0521			
MnO_2	.1144	.0558	MnO	.....	Pickard
	.1052	.0515			
	.0742	.0368			
Tungstic oxide, WO_3	.0197	.0200	W	2	Authors
	.0233	.0223			
H_2WO_4	.0289	.0277			
	.0329	.0322			
Chromic oxide, Cr_2O_3	.0863	.0015		4	Do
Chromic oxide and steel	.0186	None	(c)		
	.0040	.0040	(c)		
	.0268	None	(c)		
	.0040	.0040	(c)		
Vanadic oxide, V_2O_3	.0120	.0042	V_2O_3	4	Do
	.0440	.0160			
Vanadic oxide and steel	.0651	.0165			
	.0040	.0040			

[a] Bureau of Standards standard sample No. 29, FeO—24.78 per cent and Fe_2O_3—52.20 per cent.

[b] Bureau of Standards standard sample No. 25, MnO_2=87.11 per cent, Fe_2O_3—0.67 per cent and H_2O—1.43 per cent.

[c] No reduction of Cr_2O_3; the water observed came from reduction of oxides in the steel.

[15] Carnegie Scholarship Memoirs, Iron and Steel Inst., 5, pp. 81–82.

Summarizing these results, it is seen that the Ledebur method determines all oxygen present in steel as FeO, Ni_2O, and WO_2 [16] or higher oxides of these metals, in oxides of manganese higher than MnO (if such can be imagined to be present in steel) and in oxides of titanium and vanadium above Ti_2O_3 and V_2O_3, respectively, but none of the oxygen present in SiO_2, Al_2O_3, or Cr_2O_3.

The reducibility by hydrogen of carbon monoxide and carbon dioxide was also investigated by Pickard. He heated known amounts of pure calcium carbonate, using the same procedure as when he determined oxygen in steel. The carbonate was thus dissociated, and the carbon dioxide liberated was subjected to the reducing action of hydrogen at 900 to 1000°, the whole mass of gas being in a confined space. Pickard thus converted 60 to 100 per cent of the oxygen present in the carbon dioxide to water. The yield of water varied according to the relative quantities of hydrogen and carbon dioxide present, the time of heating, and other factors.

We have repeated Pickard's work under our own conditions for carrying out the Ledebur method, viz, heating in a current of hydrogen instead of in contact with a definite amount of that gas in a confined space. The percentages of unreduced carbon dioxide in four such experiments were, respectively, 57.0, 56.0, 56.0, and 25.0.

From Pickard's results and our own we concluded that the reducibility of carbon monoxide and dioxide would vary according to conditions, and a separate research was carried out to fix these conditions somewhat more definitely. The results of this work will be given in a paper soon to be published on Equilibria Between Carbon, Hydrogen, and Iron Oxide, by J. R. Cain and Leon Adler.

This paper shows that, in order to obtain the maximum yield of water when carbon, hydrogen, and oxide of iron are heated together, i. e., the conditions in the determination of oxygen in steel by the Ledebur method, it is necessary to pass the hydrogen at a rate of not less than 60 cc per minute. Under such conditions not less than 75 to 80 per cent of the oxygen present as iron oxide and oxides of manganese above MnO is obtained as water. At slow rates of passage of the hydrogen, diminishing amounts of water are obtained. At 5 cc per minute, for instance, only 22.5 per cent of the oxygen was given off as water. The balance was liberated as CO and CO_2.

[16] Undoubtedly Cu_2O should be included here also.

Reference should be made here to the attempts that have been made by others to determine the oxygen escaping as carbon monoxide along with the hydrogen that passes the phosphorus pentoxide tube during Ledebur oxygen determinations. A method sometimes used was to pass these gases over heated iodine pentoxide with the object of oxidizing the carbon monoxide and determining its amount either by measuring the equivalent carbon dioxide or iodine formed during the reaction. Such methods we find to be subject to large errors, for the iodine pentoxide not only oxidizes the carbon monoxide that may be present, but it also oxidizes certain hydrocarbon gases that are always formed by interaction of the hydrogen and the carbon in the steel. Such hydrocarbon gases are the result of secondary reactions between steel and hydrogen, and do not correspond to any gases present in the steel originally. These may, however, cause large errors in the determination of carbon monoxide by the iodine pentoxide method.

A method is being studied at this Bureau for correctly determining the carbon monoxide present as such in steels or generated by reaction of oxides and the carbon present in the steel upon heating it. This work will be described in a subsequent paper. The determination of the carbon monoxide present in steel and its differentiation from that generated by the action of carbon or carbides and heat on the other oxides present are subjects of considerable importance to metallurgists and will be discussed fully in this subsequent paper.

VI. DETAILS OF METHOD USED FOR DETERMINING OXYGEN

Twenty to thirty grams of sample cut under oil, washed with benzol, dried, and preserved in a desiccator, all as described on pages 18 to 25, are weighed and immediately transferred to the quartz boat, which is kept in a desiccator when not in use. The boat is inserted into the cold combustion tube from end *K* (Fig. 3). The cap is replaced at *O*, the weighed phosphorus pentoxide tubes connected, and the passage of hydrogen through the combustion tube at not less than 3.6 liters per hour begun by opening cock *H* (Fig. 1). Previous to this the generator and apparatus up to cock *L* should have been freed from air as shown by the test described on page 14. As soon as absence of oxygen is thus indicated, cock *L* is opened, the catalyzer is heated to working temperature, and the hydrogen is passed for an hour. The heating of

the furnace is then begun. This heating should be pushed quite rapidly by cutting out most of the resistance in series with the furnace. The passage of hydrogen is continued at the same rate and the temperature is held at 1000° for an hour. At the end of this period the action is over. Cocks *K* and *L* on the combustion tube and the cocks on the U tube are then closed, the hydrogen current by-passed or stopped, and the U tube detached from the furnace and immediately weighed. This weight may be recorded for information, if desired, but the weight after one-half hour's standing in the balance case is the one to be used. A counterpoise is employed, as already stated. The percentage of oxygen in the sample is calculated from the observed weights after deducting the blank obtained as already directed. The furnace may be cooled for the next determination by opening the hinged portions, the current being left on all the time.

Table 9 shows how closely results by this method will check when samples of uniform size of chip are used. These are Bureau of Standards analyzed samples, in which all particles are very uniform in size, as is well known. The only purpose in inserting this table is to show how closely duplicates by the Ledebur method, as we use it, can agree, namely, within 0.002 per cent oxygen. The actual percentages given in the table are erroneous, because of surface oxidation and too coarse chips.

TABLE 9.—Oxygen Results Using Bureau of Standards Analyzed Steel Samples.

No. 1, Bessemer 0.8, per cent oxygen found	No. 2, Bessemer 0.4 (10B), per cent oxygen found	No. 3, Bessemer 0.1 (8A), per cent oxygen found
0.012	0.025	0.031
.011	.026	.030
.011	.025	.030
.011	.....	.031
.010	.....	.029
.012	.....	.....
.010	.....	.....
.012	.....	.....
Av. 0.011	.025	.030
[a] 0.001	[a] .001	[a] .001

[a] Greatest variation from average.

Nos. 1, 2, and 3, in Table 9, correspond to Nos. 23, 10b, and 8a of Table 10, respectively. Samples in Table 10 were prepared from the original bars used in making the corresponding Bureau of Standards analyzed samples. The procedure in making chips from

these bars was that described on pages 20 to 24. The averages in Table 10 represent the true Ledebur oxygen content of these samples within the probable experimental error of the method, which, as stated, is 0.002 per cent oxygen.

TABLE 10.—Authors' Determination of Ledebur Oxygen Content of Steels and Pig Iron

[The open-hearth Bessemer and special steel samples are milled from the bars used for making Bureau of Standards standardized samples.]

Kind of steel	Bureau of Standards No.—	Carbon	Oxygen
		Per cent	Per cent
A. Acid open-hearth	34	0.84	0.010
	21A	.62	.012
	20A	.39	.021
	19A	.20	.023
	18	.10	.015
B. Basic open-hearth	14A	.81	.006
	13A	.58	.016
	12A	.36	.012
	11A	.22	.014
	15A	.11	.018
C. Acid Bessemer	23	.80	.014
	22	.59	.020
	10b	.38	.014
	9a	.25	.014
	8a	.08	.021
D. Special steels	[a] 31	.....	.011
	[b] 32	.....	.005
	[c] 33	.....	.011
	[d] 30	.....	.010
E. Successive ingots of a duplex process steel, i. e., Bessemer open-hearth	.....	.....	0.016
			.014
			.014
			.014
F. Crucible steel	.....	.....	.009
G. Electric furnace steels—1	.....	.....	.010
2	.....	.....	.007
3	.....	.....	.006
H. Johnson pig iron: [e]			
Coarse portion	.....	.....	.014
			.013
			.014
Fine portion	.....	.....	.110
			.116
			.122

[a] Chrome tungsten.
[b] Chrome nickel.
[c] Nickel.
[d] Chrome vanadium.
[e] Upon washing with benzene after milling under oil, as described on page 21, there was a separation of the milled sample into two layers. These layers were separated by decantation; the upper carried the fine portions of the samples (about 10 per cent by weight). The material in suspension in each case was separated by filtration and dried as described. The oxygen is evidently much segregated in the fine portions of these irons.

GENERAL CONCLUSIONS

From our investigations it is evident that the Ledebur method for determining oxygen in steel and iron is subject to many errors not hitherto fully recognized, and that it is very difficult to avoid such errors and to obtain correct determinations by this method. In fact, the precautions required are so great that the method can hardly be regarded as practicable for use as a control method in steel works even if it yielded information of value for control, which is doubtful, as will be shown.

Even when correct Ledebur determinations are made, the only information thus obtained is the content of oxides, existing as such, of iron, manganese (above MnO), and of copper, nickel, and tungsten, if steel ever contains unreduced oxides of the latter three metals, which seems unlikely. The Ledebur method as ordinarily practiced gives little or no information about the oxides of carbon present in steel, and there are good reasons for believing that the oxygen so combined is of great importance.

An inspection of the results in Table 10 shows that there are no characteristic features in respect to Ledebur oxygen content among all the classes of material we have examined. The results to be communicated in a later paper giving Ledebur oxygen determinations on steels deoxidized with ferrosilicon, ferrotitanium, aluminum, ferromanganese, and with a combination of ferrosilicon and ferromanganese, show no connection between Ledebur oxygen determinations and methods of deoxidation. Our own work and Pickard's show that the Ledebur method does not determine any oxygen left as manganous oxide, and it could not therefore determine any residual manganous silicate left in the steel as a result of deoxidation with manganese. The work of Kassel [17] shows that only the ferric iron of iron silicate slags is reduced under the conditions of the Ledebur method. Silicate slags containing ferric oxide are not at all likely to be present in deoxidized steels, the iron in these silicates being present in the ferrous form only. Hence the Ledebur method fails even to determine the oxide of iron present as impurity in steel if there is enough silica present in the bath to combine with it, as is nearly always the case.

From this discussion it appears then that: (*a*) The Ledebur method requires extraordinary precautions to obtain reliable

[17] Beiträge zur Kenntniss der Reduktion von Eisenschlacken durch Kohlenoxyd und Wasserstoff, Dissertation, Berlin, 1906.

results. The errors we have described undoubtedly affect in greater or less degree nearly all results by this method that have been described in the literature, and if these are approximately correct, it is because of compensating errors; (*b*) the Ledebur method as described herein determines with certainty only oxide of iron, oxides of manganese above MnO, and the oxides of nickel, copper and tungsten, if the latter three ever exist in steel. In turn, such oxides are determined correctly only in case they exist in the metal uncombined as silicates, which probably seldom happens; (*c*) Ledebur oxygen determinations show no distinguishing features for acid Bessemer, basic or acid open-hearth, duplex, electric furnace, or crucible steels; (*d*) Ledebur oxygen determinations show no differences in steels deoxidized with a variety of deoxidizers.

In view of these facts we believe that the method is of little practical value, and that it must be superseded by others which are more positive and more specific in the information they yield.

VII. SUMMARY

1. The forms in which oxygen may occur in steel and the extent to which the Ledebur method determines these are discussed.

2. Sources of error in manipulation and use of the Ledebur apparatus are described with suggestions for minimizing or eliminating such errors.

3. The suitability of various kinds of combustion tubes, special hydrogen generators, boats, and weighing tubes is considered.

4. A new type of electric furnace suitable for Ledebur determinations is shown.

5. Errors from faulty sampling of metal, too large chips, discarding fine portions of sample, and cutting in air are described and a method is given for cutting chips under oil so as to prevent large errors from surface oxidation.

6. A method of carrying out the Ledebur procedure is described with a view to eliminating most of the errors described.

7. Results by this method are given for Bessemer, open-hearth, duplex (Bessemer basic open-hearth), electric furnace, and crucible steels and for special pig iron—all of American manufacture.

8. The specific application of the Ledebur method and its limited practical use are discussed.

WASHINGTON, August 23, 1918.

DEPARTMENT OF COMMERCE

TECHNOLOGIC PAPERS

OF THE

BUREAU OF STANDARDS

S. W. STRATTON, DIRECTOR

No. 119

THE ULTRA-VIOLET AND VISIBLE TRANSMISSION OF EYE-PROTECTIVE GLASSES

BY

K. S. GIBSON, Assistant Physicist

and

H. J. McNICHOLAS, Assistant Physicist

Bureau of Standards

ISSUED JUNE 7, 1919

PRICE, 10 CENTS

Sold only by the Superintendent of Documents, Government Printing Office
Washington, D. C.

WASHINGTON
GOVERNMENT PRINTING OFFICE
1919

PREFACE

Many glasses have been recommended and extensively advertised to absorb injurious radiant energy and so protect the eyes when worn as spectacles or goggles. However, physiologists and oculists desiring to experiment to determine the value of such glasses have little to guide them in their selections except the claims of makers and sales agents. In order to experiment or prescribe intelligently it is obviously necessary that authentic and authoritative data should be available on the spectral transmission of the various glasses which are being commercially promoted as valuable in protecting the eye from harmful radiant energy.

Feeling this need of data, Dr. W. C. Posey, chairman of the committee on hygiene of the eye, American Medical Association, formally requested this Bureau to make an investigation of the spectral transmission of these glasses. The present paper is a report upon the investigation undertaken in response to this request. It deals only with the visible and ultra-violet transmissions, an analogous paper, dealing with infra-red transmissions, having already been published by the Bureau.[1]

Part of the determinations have been made in the physical laboratory at Cornell University. Thanks are due to Prof. E. L. Nichols, of Cornell, for courtesies extended the Bureau in affording facilities to expedite this work, which would otherwise have been greatly delayed.

The authors have had the advice and cooperation of Irwin G. Priest who originally planned and initiated this investigation, but was obliged to lay it aside on account of other duties.

J. T. Filgate has assisted in observing and computing.

[1] Coblentz and Emerson, Bur Standards Tech. Paper No. 93, May, 1917; 2d edition, April, 1918; 3d edition, February, 1919.

THE ULTRA-VIOLET AND VISIBLE TRANSMISSION OF EYE-PROTECTIVE GLASSES

By K. S. Gibson and H. J. McNicholas

CONTENTS

	Page
I. Introduction	3
II. Brief elementary discussion for those unfamiliar with spectrophotometry	5
1. Explanation of terms used in this paper	5
2. Illustration of method of computing the transmission for thicknesses different from that measured	9
III. Instruments used	12
IV. Specimens studied	13
V. Results of the investigation	16
1. Transmission curves	16
2. Total transmission factors	18
VI. Methods, agreements, and accuracy attained	18
1. Hilger apparatus for ultra-violet	18
2. Lummer-Brodhun spectrophotometer	19
3. Koenig-Martens spectrophotometer	20
4. Martens photometer	21
5. Agreements by the different methods	21
6. Estimated accuracy of the transmission curves	22
VII. Summary	22

I. INTRODUCTION

It is well known that ultra-violet radiant energy of certain wave lengths, such as occur in the quartz-mercury arc, the iron arc, and many other sources, is extremely injurious to the eye. Excessive brightness in the visible spectrum may also cause temporary or permanent injury; while the intense heat or infra-red radiant energy coming from molten glass or metal is also considered by some as being dangerous to the eye.

This danger of injury from such sources undoubtedly depends on both the wave length and the intensity of the radiant energy, but in spite of the great amount of work which has been done on this subject [2] quantitative data with few exceptions are not available. It is only recently that accurate data on the transmission

[2] Report of research committee, Trans. I. E. S., 9, p. 307; 1914. W. E. Burge, Trans. I. E. S., 10 [2], p. 932; 1915.

of eye-protective glasses have begun to be published. The following list comprises all the important previous publications which we have found giving data on this subject.

In 1911 the Bureau published the spectral transmissions of a few glasses of well-known trade names as determined by P. G. Nutting at the Bureau.[3]

In 1914 Crookes [4] published the results of an investigation in which he made and tested over 300 specimens in the search for the ideal colorless or neutral glass which should absorb all the ultra-violet and infra-red. Though he failed to find this ideal glass, he did succeed in producing a colorless glass which absorbed much more of the ultra-violet than ordinary colorless glass, and a blue-green glass absorbing about 90 per cent of the radiant heat. In his paper he gives, for plates of each glass 2 mm thick, the "limit of transmission of ultra-violet," the "percentage of heat cut off," the "percentage of luminous rays transmitted," and "the color as registered on a Lovibond tintometer"; but no data are given as to the per cent transmission at different wave lengths.

About the same time Luckiesh [5] measured photographically the per cent transmission from 300 to 500 mμ of nine samples of eye-protective glass—one colorless lead glass, three "smoke" or neutral, one amethyst, two amber, one "Euphos" and one "Akopos"—without, however, giving the thickness. A number of spectrograms of these and other eye-protective glasses are also given, showing approximately the limit of transmission in the ultra-violet, but again the thickness is usually not indicated. The total transmission factors for the visible are given.

In a paper on colored glasses Gage [6] compares the colorless "Crookes" glass with the "Noviol" glass manufactured by the Corning Glass Works. Transmission curves and spectrograms, 400–720 mμ, of "Noviol," "Noglare," "Noviweld," heat-absorbing and railway-signal glasses are given.

A paper by Martin [7] has appeared during the progress of this investigation which gives quantitative data for ultra-violet, visible, and infra-red transmission of various shades of neutral, "Crookes" neutral and green shades, "Fieuzal" and "Hallauer" glasses. The thickness is given in all cases. He made use of the Hilger sector photometer to obtain his ultra-violet data.

---

[3] Bur. Standards Circular No. 28, p. 13; March, 1911.
[4] Phil. Trans. Royal Soc., A 214, p. 1; 1914.
[5] Trans. I. E. S., 9, p. 472; 1914.
[6] Trans. I. E. S., 11, p. 1050; 1916.
[7] Trans. Opt. Soc., XVIII; April, 1917.

Coblentz and Emerson[8] have published considerable data on the infra-red transmission of eye-protective and other glasses. The samples studied included yellow-colored, "Crookes" neutral and green glasses, various glasses for absorbing the infra-red, such as gold-plated, blue-green (heat absorbing), greenish-brown, black, and "Noviweld" glasses, the thickness being given in all cases. They have also given the per cent transmission of these samples for the undispersed radiant energy from the gas-filled tungsten lamp, the quartz-mercury arc, and the magnetite arc.

Only the last of these papers supplies extensive data such as are presented herewith for glasses at present on the American market. In some cases comparison of data in this paper with previous data will indicate the degree of agreement obtaining in the spectral transmissions of accidentally selected glasses bearing the same trade name.

II. BRIEF ELEMENTARY DISCUSSION FOR THOSE UNFAMILIAR WITH SPECTROPHOTOMETRY

1. EXPLANATION OF TERMS USED IN THIS PAPER

Radiation is defined as the transference of energy from one point to another by means of vibratory motion in an intervening medium. This medium is caused to vibrate by a source which sends waves out through it with a certain velocity, the number of waves passing a given point per second, or the frequency, being in general the same as the number of vibrations per second of the vibrating source. The greater the frequency the shorter the wave length and vice versa, the relation being given by the formula

$$v = f\,\lambda,$$

where v is the velocity of the waves in centimeters per second, f is the frequency or number of waves passing a given point per second, and λ is the wave length in centimeters. If a suitable receiver is placed in its path, the energy may be detected.

Light, radiant heat, the radiant energy used in wireless telegraphy, ultra-violet radiant energy, and X rays are all forms of radiant energy which, though seemingly greatly different, are essentially alike. They are all generated by some form of electrical oscillations, whether they be vibrations in an electrical circuit or vibrations of atoms or electrons. They are propagated through space as waves by the same medium, the ether, and at the

[8] Loc. cit.

same velocity, approximately 300 000 000 meters (186 000 miles) per second. They differ only as regards their frequencies and the resulting wave lengths.

"Wireless" radiant energy is generated by oscillatory currents in an electrical circuit. It may be detected by other electrical circuits which are properly tuned to vibrate with the same frequency as that of the incident waves. X rays are produced by the bombardment of a piece of metal by electrons moving with high velocities in a vacuum tube. They may be detected by a fluorescent screen or by the photographic plate.

Radiant heat, light, and ultra-violet radiant energy are produced in various ways and usually all at the same time, though in greatly different relative and absolute amounts from different sources. In order to study these forms of radiant energy intelligently, it is desirable to separate from one another those of different wave lengths. They do not travel through material medialike glass with the same velocity as they do through space, but in general with smaller velocity, and the shorter the wave length the smaller the velocity of the radiant energy through the medium. The ratio of the velocity in one medium to that in a second medium is called the refractive index from the first medium to the second. When radiant energy of different wave lengths, such as is emitted from incandescent sources like the sun, the tungsten electric lamp, or the gas flame, is passed through a prism of glass, quartz, or other transparent material, this difference in velocities results in the direction being changed; that is, it is deviated, and in general the shorter the wave length the greater the deviation. If, after passing through the prism, the radiant energy is then focused on a white screen, a spectrum is formed. To the eye this spectrum seems to be composed of the colors red, orange, yellow, green, blue, and violet in that order, the red being deviated least and the violet most. Therefore, the red light is of longer wave length than the orange, the orange than the yellow, etc.

But this is not all. If a thermometer or other sensitive heat-measuring device is placed beyond the red end of the visible spectrum, it will indicate by its rise in temperature that heat waves are being radiated to it, though the eye perceives nothing. This is called infra-red radiant energy or radiant heat and is of longer wave length than red light, since it is deviated less

Also, if a photograph be taken of this spectrum, it will be found that radiant energy of shorter wave length than the violet light is recorded by the plate, although the eye can not perceive it. This is called ultra-violet radiant energy.

Various units are used to specify wave lengths, depending on the magnitudes involved. The following are in common use, all being derived from the standard of length, the meter (1.093 yards), which is represented by the symbol *m:*

Units	Symbol
1 m = 0.001 kilometer	km
1 m = 1000 millimeters	mm
1 mm = 1000 microns	μ
1 μ = 1000 millimicrons	$m\mu$ [a]
1 $m\mu$ = 10 Ångström units	Å

[a] The symbol $m\mu$, to represent the millimicron, is used on the following authorities: C. E. Guillame, Unités et Étalons, p. 7; 1893. Société Française de Physique, Recueil de Constantes Physiques, p. 1; 1913. The millimicron has been commonly represented by the symbol $\mu\mu$, which symbol has now been discarded as being inconsistent with the metric system, the micron μ denoting one millionth.

In the following table is shown the approximate range of wave lengths included in the various forms of radiant energy:

Form	Range of wave length
"Wireless"	12 km ($12 \times 10^{12} m\mu$) — 1.6 mm (1600μ, 1600000 $m\mu$)
Radiant heat or infra-red	310 μ (310000 $m\mu$) — 0.77 μ (770 $m\mu$)
Light or visible	770 $m\mu$ — 380 $m\mu$
Ultra-violet	380 $m\mu$ — 60 $m\mu$ (600Å)
X rays or Roentgen	12 Å (1.2 $m\mu$) — 0.2 Å (0.02 $m\mu$)

It should be understood very clearly that the only difference between the infra-red, the visible, and the ultra-violet radiant energy is that of frequency and wave length. There is no gap in the spectrum between these three forms as there is between "wireless" radiant energy and radiant heat, or as still exists between ultra-violet radiant energy and X rays. The only reason that this classification into infra-red, visible, and ultra-violet exists is because it so happens that the eye is sensitive to radiant energy of a certain range of wave lengths in the middle part of the spectrum. If a source could be found which emitted equal amounts of energy at all wave lengths, then a blackened thermometer or radiometer placed in its spectrum would be affected the same at all wave lengths. It could detect no difference

between ultra-violet of wave length 290 mμ, green light of 540 mμ, or infra-red of 6000 mμ. It would be equally sensitive to them all.

If this equal-energy spectrum were photographed, however, no trace of the red or infra-red radiant energy would be found, unless the plate had been specially sensitized. Green light would be recorded slightly, but the blue, violet, and ultra-violet would affect the plate very easily; that is, the photographic plate has a varying spectral sensitivity.

Now, if this equal-energy spectrum were viewed by the human eye, a still further difference would be noted. Like the photographic plate, the eye is sensitive to radiant energy of certain wave lengths (much different from the plate), which is thus made visible and called light; but by definition the eye can not perceive the ultra-violet or infra-red. But unlike the plate or the radiometer, the eye can perceive a difference in the radiant energy of different wave lengths to which it is sensitive, provided these wave lengths are not too nearly the same; that is, the eye can distinguish light of different wave lengths by its hue—red, green, etc. It can perceive green light easier than that of any other hue, blue and yellow less easily, violet and red least of all, and where the violet and red are no longer perceptible the ultra-violet and infra-red are said to begin. These boundaries are thus somewhat vaguely defined, varying with conditions such as brightness, proximity of other light, different eyes, etc.

This relative sensibility of the eye to light of different wave lengths has been accurately studied. In Fig. 1 (and also Figs. 2–20) the values[1] have been plotted in the form of a curve. This curve is known as the visibility curve of the average human eye for an equal-energy spectrum. It is somewhat different for different observers, and the maximum is shifted to shorter wave lengths for light of very low intensity.

The amount of energy emitted per second, or the radiant power, is well known for many sources. Some sources, like incandescent solids and liquids, where the radiant power is due primarily to temperature, give what is known as a continuous spectrum; that is, all wave lengths over a considerable range are present. The relative spectral distribution of radiant power in a number of such sources is shown in Fig. 1. The radiant powers have all been reduced to 100 at 590 mμ. The maximum radiant power for most

[1] Coblentz and Emerson, Bur Standards Scientific Papers, No. 303; September, 1917.

of these sources is in the infra-red; but the higher the temperature the shorter the wave length of the maximum until at 5000–6000° C (which is probably about the temperature of the sun) the maximum lies in the visible. The radiant power for all these sources continues to decrease as the ultra-violet region is entered and sooner or later becomes insignificant.

Other sources like the electric arc or spark, in air or in a vacuum, do not in general give a continuous spectrum, but instead the spectrum appears as a number of sharp lines separated by dark regions. Such spectra are termed line spectra and are apt to be very rich in ultra-violet. Each element has its own particular line spectrum different from that of every other element. In Fig. 1 is shown the relative radiant power of the light from the mercury arc, reduced to 100 at 578 mμ. Some of the lines, though plotted as single, are made up of two or more components whose wave lengths are so nearly the same that the radiant power of the group as a whole was of necessity measured.

If the relative radiant power of a source at certain wave lengths is multiplied by the relative visibility of the average human eye at the same wave lengths, a new curve will be obtained which is called the luminosity curve for that source. Like the visibility curve, the luminosity curve for any source is zero in the ultra-violet and infra-red. In fact, the visibility curve is simply the luminosity curve of a source emitting equal amounts of energy at all wave lengths in the visible spectrum. The luminosity curves for the Hefner lamp and blue sky have been thus computed and plotted in Fig. 1. The luminosity "curve" for the mercury arc would consist of a number of vertical lines.

2. ILLUSTRATION OF METHOD FOR COMPUTING THE TRANSMISSION FOR THICKNESSES DIFFERENT FROM THAT MEASURED

If radiant power from any source is passed through media, such as quartz, glass, solutions, etc., the relative spectral distribution may be greatly altered, part of the energy being reflected, part absorbed, and the rest transmitted. The amount absorbed or transmitted may vary greatly with the wave length, but throughout the visible and ultra-violet the amount reflected remains nearly constant. The following brief discussion will make clear the relations existing between reflection, transmission, and absorption, and illustrate the method used in computing the spectral transmissions for thicknesses other than that measured.

Let radiant power be incident normally upon a plate of glass or other material of thickness b with plane parallel surfaces.[10] Let P_1 be the radiant power incident on the first surface, P_I that transmitted by the first surface, P_2 that incident on the second surface, and P_{II} that transmitted by the second surface. At each surface a certain fraction R of the incident power is reflected, this fraction depending on the refractive index n between the two media bounding the surface, i. e.,

$$R=\frac{P_1-P_I}{P_1}=\frac{P_2-P_{II}}{P_2}=\left(\frac{n-1}{n+1}\right)^2.$$

The transmission T is defined as the fraction of incident radiant power which gets through the specimen, i. e.,

$$T=\frac{P_{II}}{P_1}$$

and the per cent transmission is 100 T. The transmittance T is defined as the fraction of radiant power transmitted by the first surface which is incident on the second surface, i. e.,

$$\mathsf{T}=\frac{P_2}{P_I}.$$

The law of the variation of transmittance with thickness b is given by the equation $\mathsf{T}=t^b$, wherein t is called the transmissivity of the material composing the specimen and may be defined as the transmittance per unit of thickness.

The relation between T, T, and t may be shown as follows:

Since $P_I=P_1\,(1-R)$

and $P_{II}=P_2\,(1-R)$

and $P_2=P_I\mathsf{T}=P_I t^b$

$\therefore$ $P_{II}=P_1\,(1-R)^2\,\mathsf{T}=P_1\,(1-R)^2 t^b$

and $\dfrac{P_{II}}{P_1}=T=(1-R)^2\,\mathsf{T}=(1-R)^2 t^b$

In general, this equation is applied only to radiant power of definite wave length. Curves plotted between T or 100 T and λ are called spectral transmission curves or, in case of 100 T, per cent transmission curves. By means of instruments called spectrophotometers (see Sec. VI), values of T may be accurately measured wave length by wave length in the ultra-violet, visible, or infra-red.

[10] The symbols used in this paper are in accord with those to be recommended in another paper now in preparation at the Bureau, A Proposed Basic Nomenclature for Use in Colorimetry, by Irwin G. Priest.

The equation $T = (1-R)^2\mathsf{T} = (1-R)^2 t^b$ may be used to compute the transmission for a specimen of different thickness from that observed, or to compute what thickness is necessary to give any desired transmission. The following will illustrate how this may be done:

As noted above, R varies very little with the wave length, being equal to

$$\left(\frac{n-1}{n+1}\right)^2$$

where n is the refractive index between air and glass. For all practical purposes, so far as the glasses in the present investigation are concerned, n may be taken equal to 1.50, making R equal to 0.04 and $(1-R)^2$ about 0.92. Thus the equation may be written

$$\frac{T}{.92} = \mathsf{T} = t^b \text{ (for a given wave length).}$$

For some other thickness b', where the transmission will be T', the transmittance T', and the transmissivity t as before, the equation will be

$$\frac{T'}{.92} = \mathsf{T}' = t^{b'}$$

By taking logarithms, the following equations are obtained:

$$\log \mathsf{T} = b \log t$$
$$\log \mathsf{T}' = b' \log t$$

From these equations the following may be derived:

(1) $$\mathsf{T}' = \mathsf{T}^{\frac{b'}{b}}$$

(2) $$\log \mathsf{T}' = \frac{b'}{b} \log \mathsf{T}$$

(3) $$b' = \frac{\log \mathsf{T}'}{\log \mathsf{T}} b$$

The examples in Table 1 will illustrate how these last equations may be used for numerical computations. The unit of thickness is taken as 1 mm. Logarithms are taken to the base 10. However, the same results will of course be obtained if logarithms to the base e and any other unit of thickness are used.

The computations shown in Table 1 may be much more easily performed graphically by use of the chart shown in Fig. 25, which is a reproduction of the one in use at the Bureau. The legend underneath explains the method of use.

TABLE 1.—Method of Computing Transmission for Different Thicknesses

GIVEN T=0.50 AND b=1.68 mm. TO FIND T′ WHEN b′ EQUALS 3.36 mm OR 2 b, 0.56 mm OR ⅓ b, 5.37 mm, OR 0.73 mm, USING EQUATIONS (1) OR (2)

Problem No.	b′ in mm	$\frac{T}{T/.92}$ = .50/.92 = .544	Log T = Log .544 = 9.736−10 = −.264	b′/b	$\text{Log } T' = \frac{b'}{b}\text{Log } T$ $= \frac{b'}{b}(-.264)$	$T' = T\frac{b'}{b}$	$T' = .92\,T'$
1	3.36 or 2 b	0.544	.....	2	.....	$0.544^{2} =$ 0.296	0.272
2	.56 or ⅓ b	.544	.....	⅓	.....	$.544^{\frac{1}{3}} =$ .817	.752
3	5.37	.544	−0.264	3.195	3.195(−0.264) = −0.844 = 9.156−10	.143	.132
4	.73	.544	− .264	.434	.434(−0.264) = −0.1145 = 9.8855−10	.768	.706

GIVEN T=0.50 AND b=1.68 mm. TO FIND b′ WHEN T′=0.30 OR 0.87 USING EQUATION (3)

Problem No.	T′	$\frac{T'}{T'/.92}$ =	Log T′	$\frac{T}{T/.92}$ =	Log T	$\frac{\text{Log } T'}{\text{Log } T}$	$\frac{b'}{\frac{\text{Log } T'}{\text{Log } T}b}$ =
5	0.30	0.326	9.513−10 = −0.487	0.544	−0.264	$\frac{-0.487}{-0.264} =$ 1.84	1.84×1.68 = 3.095
6	.87	.945	9.976−10 = −0.024	.544	− .264	$\frac{-0.024}{-0.264} =$ 0.091	0.091×1.68 = 0.153

III. INSTRUMENTS USED

The data presented in this paper have been obtained partly at this Bureau and partly in the physical laboratory at Cornell University. The determinations by the Hilger sector apparatus and the Lummer-Brodhun spectrophotometer were made at Cor-

nell,[11] those by the Koenig-Martens spectrophotometer and the Martens photometer at the Bureau.

In Table 2, under the heading "Instrument" is indicated for each specimen by an asterisk (*) which instruments were used. With the Hilger apparatus wave lengths were determined photographically in the range 230–500 mμ for per cent transmissions between 1–5 and 100. On the Lummer-Brodhun and Koenig-Martens spectrophotometers, values of the per cent transmission could be obtained from 430 to 710 mμ. In Section VI is given an account of the method of use of the instruments, the agreements between methods, and the estimated accuracy of the results.

IV. SPECIMENS STUDIED

The following dealers or manufacturers have kindly donated samples to the Bureau for examination in this investigation:

Company	Referred to in report as—	Address	Number of samples
American Optical Co.	A. O. Co.	Southbridge, Mass.	47
Chicago Eye Shield Co.	C. E. S. Co.	2300–2304 Warren Ave., Chicago, Ill.	6
Corning Glass Works.	Corning.	Corning, N. Y.	13
F. A. Hardy & Co.	Hardy.	10 South Wabash Ave., Chicago, Ill.	2
Julius King Optical Co.	King.	12 Maiden Lane, New York.	5
Wall & Ochs (through Dr. W. C. Posey).	W. & O.	Philadelphia, Pa.	9

These specimens[12] are all designated by their trade names. Some of them are of unknown origin, specimens which have been marketed for years under more or less well-recognized trade names. The data given show what may be obtained under one of these names, but, as there are usually so many different shades of the same kind of glass marketed under the single trade name, it can not be expected that duplicates can be obtained merely by ordering by name. However, most of the specimens, especially those of American manufacture, are designated by shade—that is, "A," "B," etc.—as well as by trade name or by number, and in this case it is expected that approximately duplicate samples can be obtained. If accurate reproduction is required, the designation should not be assumed as a sufficient specifica-

[11] Apparatus for the determination of spectral transmissions by this photographic method as well as by the photoelectric (null method) has since been installed at the Bureau, and such determinations are now made here.

[12] It is hoped later to supplement this paper with another giving data on other kinds of eye-protective glasses at present on the market.

tion; but the oculist or buyer, guided by the data given herewith, may now prescribe or order glasses of specified spectral transmissions, acceptance being subject to test.

The surfaces of these specimens were highly polished and with very few exceptions made plane and parallel to 0.01 mm. They are of a thickness ordinarily used in spectacle lenses. They have been labeled and filed at the Bureau for future reference. In Table 2 is given a list of these glasses arranged alphabetically according to their trade names, together with the name of the company, the color, thickness, and other data which will be discussed later.

TABLE 2.—List of Specimens Studied

Specimen, company's designation	Company	Color	Thickness in millimeters	Data, Fig. No. —	Instrument[a]				Total transmission factor
					H	L-B	K-M	M	
Akopos	King	Yellow-green	2.17	13	(*)	(*)	.....	.....	0.36
Amber A	A. O. Co	Yellow	2.13	10	(*)	.....	(*)	(*)	.79
Amber B	do	do	2.13	10	(*)	.....	(*)	.....	.57
Amber dark	W. & O	do	1.85	10	(*)	(*)	(*)	(*)	.555
Amber light	do	do	1.93	10	(*)	(*)	(*)	(*)	.805
Amethyst A	A. O. Co	Purple	2.08	15	(*)	.....	(*)	.....	.745
Amethyst B	do	do	2.05	15	(*)	.....	(*)	(*)	.70
Amethyst C	do	do	2.04	15	(*)	.....	(*)	.....	.55
Amethyst dark	W. & O	do	1.90	16	(*)	(*)	(*)	(*)	.705
Amethyst light	do	do	1.97	16	(*)	(*)	(*)	(*)	.805
Arkweld dark	King	do[b]	2.46	21	(*)	.....	.....	.....	0+
Blue A	A. O. Co	Blue	2.10	17	(*)	.....	(*)	.....	.88
Blue B	do	do	2.04	17	(*)	.....	(*)	.....	.62
Blue C	do	do	2.05	17	(*)	.....	(*)	.....	.35
Blue D	do	do	2.11	17	(*)	.....	(*)	.....	.062
Chlorophile	W. & O	Yellow-green	1.98	13, 23	(*)	(*)	(*)	(*)	.65
Chromatic test	A. O. Co	Blue	2.36	18	(*)	.....	(*)	.....	.0075
Cobalt blue AA	do	do	2.75	18, 24	(*)	.....	(*)	.....	.033
Cobalt blue A	do	do	3.20	18	(*)	.....	(*)	(*)	.026
Cobalt blue B	do	do	1.85	18, 24	(*)	.....	(*)	.....	.0085
Cobalt blue C	do	do	1.46	18	(*)	.....	(*)	(*)	.0096
Cobalt blue D	do	do	1.86	18, 24	(*)	.....	(*)	.....	.0055
Crookes A	W. & O	Neutral	1.79	4	(*)	(*)	(*)	(*)	.84
Crookes B	do	do	1.93	4	(*)	(*)	(*)	(*)	.485
Crookes A, Wellsworth	A. O. Co	do	2.05	3	(*)	.....	(*)	.....	.85
Crookes B, Wellsworth	do	do	2.16	3	(*)	.....	(*)	.....	.45
Crown 1.50	do	do	1.68	4	(*)	.....	(*)	(*)	.92
Electric smoke red	do	Red-purple[b]	1.82	21	(*)	.....	.....	(*)	0+
Electric smoke Y	do	Yellow[b]	2.01	.....	(†)	.....	.....	.....	0+
Euphos	do	Yellow-green	1.95	11, 24	(*)	.....	(*)	.....	.72
Fleuzal	W. & O	do	1.98	12, 13, 23	(*)	(*)	(*)	(*)	.835
Fleuzal A	A. O. Co	do	2.13	12	(*)	.....	(*)	.....	.665
Fleuzal B	do	do	2.13	12	(*)	.....	(*)	.....	.51

[a] H—Hilger, L-B—Lummer-Brodhun, K-M—Koenig-Martens, M—Martens. An asterisk (*) indicates which instruments were used. A dagger (†) instead of an asterisk means that no transmission was [illegible] millimeters.

[b] Color may fade.

TABLE 2—Continued

Specimen, company's designation	Company	Color	Thickness in millimeters	Data, Fig. No. —	Instrument [a]				Total transmission factor
					H	L-B	K-M	M	
Hallauer	W. & O.	Yellow-green	1.90	13, 23	(*)	(*)	(*)	(*)	0.48
Laboratory No. 57	A. O. Co.	Neutral	1.90	6	(*)	.....	(*)	(*)	.91
Laboratory No. 58	do.	Yellow	2.02	6	(*)	.....	(*)	(*)	.815
Laboratory No. 59	do.	Blue-green	2.13	14, 24	(*)	.....	(*)	.....	.32
Laboratory No. 61	do.	Yellow-green	2.13	11, 24	(*)	.....	(*)	.....	.65
Luxfel	do.	Yellow	2.00	6	(*)	.....	(*)	(*)	.835
Noviol AA	do.	do.	1.88	9	(*)	.....	(*)	(*)	.88
Noviol A	do.	do.	2.06	9	(*)	.....	(*)	.....	.87
Noviol B	do.	do.	2.10	9, 24	(*)	.....	(*)	.....	.85
Noviol C	do.	do.	2.10	9	.....	.....	(*)	.....	.83
Noviol O	do.	do.	2.03	9, 24	(*)	.....	(*)	.....	.85
Noviol A	Corning	do.	2.00	8, 24	(*)	(*)	.....	(*)	.865
Noviol A_1	do.	do.	2.88	7	(*)	(*)	.....	.....	.87
Noviol A_2	do.	do.	2.84	7	(*)	(*)	.....	.....	.85
Noviol B	do.	do.	1.95	8	(*)	(*)	.....	(*)	.84
Noviol B_1	do.	do.	2.83	7	(*)	(*)	.....	.....	.845
Noviol C	do.	do.	2.00	8, 24	(*)	(*)	.....	(*)	.825
Noviol O	do.	do.	2.01	8	(*)	(*)	.....	(*)	.865
Noviol O	Hardy	do.	1.94	7	(*)	(*)	.....	(*)	.88
Noviweld 4	A. O. Co.	do.	1.89	20	(†)	.....	(*)	(*)	.0315
Noviweld 5	do.	do.	2.16	20	(†)	.....	(*)	(*)	.014
Noviweld 6	do.	do.	2.20	20	(-)	.....	(*)	(*)	.010
Noviweld 7	do.	do.	1.90	20	(†)	.....	.....	(*)	.006
Noviweld 8	do.	do.	2.01	20	(†)	.....	.....	(*)	.0032
Noviweld 12	do.	do. [b]	2.08	.....	(†)	.....	.....	.....	0+
Pfund	do.	Green	.60	14	(*)	.....	(*)	(*)	.047
Rifleite	King	Yellow	3.14	7	(*)	(*)	.....	.....	.775
Saniweld dark	do.	do.	2.12	13, 21	(†)	(*)	.....	(*)	.0135
Saniweld light	do.	do.	1.82	13	(*)	(*)	.....	(*)	.115
Smoke A	A. O. Co.	Neutral	2.10	5	(*)	.....	(*)	(*)	.75
Smoke B	do.	do.	2.14	5	(*)	.....	(*)	(*)	.38
Smoke C	do.	do.	2.13	5	(*)	.....	(*)	(*)	.265
Smoke D	do.	do.	2.03	5	(*)	.....	(*)	(*)	.092
Special Noviweld No. 8	Hardy	Yellow [b]	1.77	22	(†)	(*)	.....	.....	.00019
Special Welders dark	A. O. Co.	Green [b]	2.54	19, 21	(*)	.....	.....	(*)	0+
Special Welders light	do.	do.	1.68	19, 21	(*)	.....	(*)	(*)	.0105
Welders smoke dark	do.	Neutral	1.42	19	(*)	.....	(*)	(*)	.056
Welding glass 1	C. E. S. Co.	Yellow	1.98	21	(†)	(*)	.....	(*)	.0034
Welding glass 2	do.	do.	2.32	20	(-)	(*)	.....	(*)	.045
Welding glass 3	do.	do.	1.50	21	(†)	(*)	.....	(*)	.00885
Welding glass 4	do.	do.	1.48	21	(†)	(*)	.....	(*)	.00885
Welding glass 5	do.	Red	1.87	21	(-)	(*)	.....	(*)	.0027
Welding glass 6	do.	Yellow	1.97	20	(-)	(*)	.....	(*)	.0705
1.52	A. O. Co.	Neutral	2.33	2	(*)	.....	(*)	(*)	.92
91 B	Corning	do.	1.97	3	(*)	(*)	(*)	(*)	.84
124 IP	do.	Green	2.09	14, 21	(*)	(*)	.....	(*)	.015
124 JA	do.	Blue-green	2.02	14, 24	(*)	(*)	.....	(*)	.61
391 DD	do.	Red [b]	1.90	22	(†)	(*)	.....	.....	.00012
392 F	do.	Blue-green	1.90	14, 24	(*)	(*)	.....	(*)	.72

[a] H=Hilger, L-B=Lummer-Brodhun, K-M=Koenig-Martens, M=Martens. An asterisk (*) indicates which instruments were used. A dagger (†) instead of an asterisk means that no transmission was detected from 380 to 800 millimicrons.

[b] Color very dense.

[c] Could film.

V. RESULTS OF THE INVESTIGATION

1. TRANSMISSION CURVES

The results of the investigation are shown by curves in Figs. 2–22, in which the per cent transmissions for different wave lengths are plotted against the wave lengths. By per cent transmission is meant that per cent of the incident radiant energy which gets through the specimen. The curves are drawn in heavy lines as far as the actual observations were carried. Estimated values are indicated by the dotted parts. In each figure (except Figs. 21 and 22) is also plotted the relative visibility curve of the average human eye for a source emitting equal amounts of energy at all wave lengths in the visible spectrum. In each figure (except Figs. 21 and 22) is also given the transmission curve of a specimen of crown glass 1.68 mm thick (specimen "Crown 1.50," Fig. 2) so that one may see at a glance in what respect the various glasses protect the eye better than ordinary colorless glass. Of course in comparing the transmission curves of two specimens, any difference in thickness must be allowed for. (See Sec. II and Fig. 25.)

Little need be said in discussion of the curves, as the information desired may be obtained by inspection of them, together with Table 2. It must be remembered that conclusions reached apply only to the particular glasses herein listed.

Of the specimens studied, the five kinds which are most efficient as protection against the ultra-violet, while being at the same time nearly colorless in the thicknesses examined, are "Crookes A" (Figs. 3–4), Corning "91B" (Fig. 3), A. O. Co. "Lab. No. 57" (Fig. 6), A. O. Co. "Lab. No. 58" (Fig. 6) and "Noviol O" (Figs. 7–9). Of these, "Noviol O" and A. O. Co. "Lab. No. 58" are the best, but are not so truly colorless as the other three. Of the slightly colored glasses, by far the best seem to be "Noviol A" (Figs. 8–9) and "Noviol A_1" (Fig. 7), as they absorb completely below 410 mμ while transmitting about 87 per cent of the incident light. It is not thought that the slight color would be at all objectionable for ordinary use.

A combination of "Noviol A" (Figs. 8–9) and Corning "124JA" (Fig. 14) is very efficient for eye protection, as it absorbs all the ultra-violet and most of the infra-red, and still has high visible transmission. The color is a very light green, and the colors of objects viewed through it are distorted practically none at all.

A gold film on "Noviol A" glass would also be very efficient, though transmitting less of the visible than the combination just mentioned.

The yellow and yellow-green glasses (Figs. 7–13) of a deeper shade are usually good protection against the ultra-violet. The green and blue-green glasses of Fig. 14 are used primarily to protect the eye from the infra-red. The "Pfund" specimen is a gold film between two pieces of what seems to be "Crookes" glass. "Smoke" (Fig. 5), amethyst (Figs. 15–16), and blue (Figs. 17–18) or purple glasses are liable to be little better than clear glass as a protection against the ultra-violet. Of the welding glasses (Figs. 19–22), yellow seems to be the safest, as the green or neutral shades are liable to have transmission bands centering near 395 mμ, which may extend a considerable distance into the ultra-violet.

In Fig. 11 is plotted the transmission curve for a specimen called "Fieuzal" obtained from a retail store. By comparison with the curves of Figs. 11 and 12, it is seen to resemble A. O. Co. "Euphos" and "Lab. No. 61" much more than the "Fieuzal" specimens from W. & O. or A. O. Co. The resemblance between W. & O. "Chlorophile" (Fig. 13) and A. O. Co. "Fieuzal A" and "Fieuzal B" (Fig. 12) is also very great, much more than that between the latter specimens and W. & O. "Fieuzal" (Fig. 12). The resemblance between "Hallauer" and "Akopos" (Fig. 13) is very striking; also that between "Noviol C" (Figs. 8–9) and "Rifleite" (Fig. 7).

As may be noted from the curves, the transmissions of many of the glasses herein examined, especially the yellow or yellow-green, decrease to less than 1 per cent somewhere from 480 to 340 mμ, depending on the specimen, and so far as can be detected do not increase at shorter wave lengths. Transmissions of probably one-tenth the lowest value measured could be detected on the photographic negatives. Therefore, when in the figures the curves are drawn as if there were zero transmission below 500 mμ, it should be understood merely that the transmission, if there be any, is less than 0.3 or 0.4 per cent if 2.5 per cent were the lowest value measured and less than 0.1 or 0.2 per cent if 1.0 per cent were measured. Specimens one-half or one-third the thickness of those examined might show slight transmission in the ultra-violet.

2. TOTAL TRANSMISSION FACTORS

In Table 2, last column, are given the total transmission factors for light of equal energy at all wave lengths. These factors were computed as follows: At every 10 mμ from 400 to 720 mμ, the value of the relative visibility of the average human eye (which is the luminosity of a source emitting equal amounts of energy at all wave lengths in the visible spectrum) was multiplied by the fraction of incident light transmitted by the specimen at that same wave length. With these values a new curve was plotted to the same scale as the visibility curve. This new curve would show the relative luminosity of the equal energy source after the light had passed through the specimen. Then the areas under these two luminosity curves were measured with a planimeter, and the ratio of these two areas gives the total transmission factors for light of equal energy at all wave lengths. Any error in the transmission curves, of course, enters into the total transmission factors. Assuming the transmission curves to be correct, the total transmission factors given in Table 2 are considered accurate to at least one unit in the second figure.

It should be understood that the total transmission factor of any specimen depends on the relative luminosity (relative visibility times relative radiant power—see Fig. 1) of the source emitting the light which is viewed through the specimen. To compute the total transmission factor for a source whose relative spectral distribution of radiant power is known, the values of the relative luminosity of the source should be used instead of the values of the relative visibility, as was done in the computations described above. The total transmission factors for sunlight do not, in general, differ much from those for a source emitting equal amounts of energy at all wave lengths, but for the tungsten lamp and other artificial sources the difference may be considerable.

VI. METHODS, AGREEMENTS, AND ACCURACY ATTAINED

1. HILGER APPARATUS FOR ULTRA-VIOLET

The Hilger sector apparatus with the Hilger quartz spectrograph, type C, was used practically unchanged as set up by H. E. Howe.[13] The slit of the spectrograph was 0.15 mm. The source was a high-tension aluminum spark under water, which gives a continuous spectrum throughout and beyond the range 500–230

[13] Phys. Rev., N. S., VIII, p. 674; 1916.

mμ. This photographic method is greatly superior to any other and compares favorably in speed and accuracy with any visual method. It consists essentially in simply determining, for any per cent transmission desired, at what wave length two contiguous spectra are of equal density; and so far as known this value, by this method, is independent of the kind, the law, the chromatic sensitivity, or the length of development of the plate, as well as independent of the kind of developer, the variations in intensity of the source, and the length of exposure. The per cent transmission is determined arbitrarily by the ratio of sector openings, which may be set anywhere from 1.00 to 0.01, the wave length being the dependent variable.

Due to certain contrast effects on the negatives, these wave lengths can be determined more accurately where the transmission curve is steep than where it is nearly horizontal, and where the per cent transmission is low than where it is high. The varying dispersion makes determinations increasingly accurate as the spectrum is traversed from 500 to 230 mμ. The method was limited at 500 mμ by the lack of sensitivity of the plates used.

Other things being equal, the uncertainty of determinations on the steep parts of the curves is 1–8 mμ from 500 to 460 mμ, 1–4 mμ from 460 to 400 mμ, and 1–2 mμ from 400 to 230 mμ. Around the peaks of transmission bands or on the parts of curves where the slope is small, this uncertainty is increased. In this as in the other methods, a number of determinations were made and the average used.

2. LUMMER-BRODHUN SPECTROPHOTOMETER

The Lummer-Brodhun spectrophotometer was used practically the same as in a previous investigation,[14] the acetylene flame, the carbon lamp, and the gas-filled tungsten lamp being used as sources, the last one being the most satisfactory. The slits of the telescope and first collimator were kept constant at 0.60 mm, while the slit of the second collimator was varied about from 0.06 to 1.00 mm. The ratio of the readings of this second slit when the specimen was placed in front of the first slit and then removed gave the fraction of incident light transmitted, except that 0.02 mm was subtracted from all readings of this second slit before computing this ratio.[15] This correction is made necessary probably by a loss of light due to reflection or diffraction at the edges of

[14] K. S. Gibson, Phys. Rev., N. S., VII, p. 141; 1916.
[15] Nichols and Merritt, Phys. Rev., XXXI, p. 593; 1910.

the slit, which is equivalent to a shift in the zero point of the screw by 0.02 mm.

When the transmission was so low as to bring the reading of the second slit below 0.06 mm and thus make the correction of 0.02 mm inaccurate, a specimen of known transmission was placed before the second slit, necessitating widening it, and making it possible to obtain values as low as 0.1 per cent with considerable accuracy. This method was used, for instance, in obtaining the curves in Fig. 21 for those specimens measured on the Lummer-Brodhun spectrophotometer.

In the case of the two specimens whose curves are shown in Fig. 22, the slits of both collimators were varied and glasses of known transmission used before them until the proper combination was obtained to make the values fairly accurate.

Except for subtracting 0.02 mm from all slit readings, no corrections were made for slit widths. In spite of the wide and varying slits used, however, the agreement with other methods shows that the error is very small except in case of a glass like "Crookes A," where the narrow bands near 580 mμ could not be accurately measured as they were on the Koenig-Martens instrument.

3. KOENIG-MARTENS SPECTROPHOTOMETER

The Koenig-Martens polarization spectrophotometer [16] was used in the usual way. Two beams of light from the same source are polarized mutually perpendicular by means of a Wollaston prism and brought together to form the two halves of a photometric field. This field is viewed through a nicol prism and the adjustment is such that the two halves of the field are brought to equality by the analyzing nicol somewhere near 45° on the scale. The specimen was placed first in one beam and then in the other, and the analyzing nicol rotated in each case until a photometric match was obtained. The tangent of the angle in one case multiplied by the cotangent in the other gives the fraction of incident light transmitted. The slit was 0.10 mm in width. A 500-watt gas-filled tungsten lamp was used as a source. Color screens were used over the eyepiece when needed to cut out stray light. On this instrument measurements were made down to 1 per cent transmission.

[16] Ann. d. Phys., Series 4, 12, p. 985; 1903.

4. MARTENS PHOTOMETER

The Martens polarization photometer,[17] working on the same principle as the Koenig-Martens spectrophotometer, but without a dispersing prism, was used with a mercury arc lamp and colored glass screens by which light of wave lengths 406, 436, and 578 mμ could be isolated. Values of transmission could be obtained accurately as low as 0.1 per cent. This method served as a valuable means of checking the other results when discrepancies appeared and also gave definite results in some cases where no other method could be used. For example, in Fig. 21 are plotted curves for specimens having weak transmission bands near 400 mμ. The wave length of maximum transmission and the approximate width of band could be obtained from the negative, also an indication as to whether or not the transmission was nearly 1 per cent. The Martens photometer gave a definite value at 406 mμ, and with these data the curves were drawn. It was also useful in giving low values at 436 mμ as in the curves of Fig. 21, and at 578 mμ as in the case of A. O. Co. "Noviweld No. 7," "Noviweld No. 8," and "Special Welders Dark."

5. AGREEMENTS BY THE DIFFERENT METHODS

In Figs. 23 and 24, a number of curves are drawn with all points plotted to indicate the kind of agreements and disagreements met with in the instruments used in this investigation. In all cases the black circles represent values obtained photographically with the Hilger sector apparatus, the white circles those obtained visually with the Lummer-Brodhun spectrophotometer, the crosses those with the Koenig-Martens spectrophotometer, and the squares those with the Martens photometer when it was used.

Between 500 and 650 mμ, values obtained by the two visual spectrophotometers seldom disagree by more than 3 units of per cent and usually agree within 1 unit, especially at the lower transmissions. Beyond 650 mμ, the discrepancies are sometimes larger, occasionally reaching 5 to 10 units around 700 mμ. Also from 500 mμ into the blue the same large discrepancies occasionally appear, especially at 440 or 450 mμ, but values obtained on the Hilger apparatus and on the Martens photometer at 406 and 436 mμ help remove doubt as to where the curve should be drawn.

[17] Phys. Zeit., I, p. 299; 1900

6. ESTIMATED ACCURACY OF THE TRANSMISSION CURVES

It is difficult to estimate the accuracy of curves of such varying types, especially when the range of wave lengths and transmission values is so great. But a careful study of these and many others measured on the same instruments indicates the following accuracies in the curves as drawn: In all figures (except Figs. 21 and 22), from 230 to 400 mμ and from 500 to 650 mμ the curves are considered accurate to 0–2 units of per cent, and from 400 to 500 mμ and from 650 to 710 mμ to 0–5 units of per cent. In Fig. 21 the curves are considered accurate to 0–3 units, the unit being 0.1 of 1 per cent, and in Fig. 22 to 0–5 units, the unit being 0.001 of 1 per cent.

VII. SUMMARY

Eighty-two samples of eye-protective glass have been studied in regard to their transmission of ultra-violet and visible radiant energy. For each specimen is given:

1. The trade name under which it is marketed,
2. The company from which it may be obtained,
3. The approximate color,
4. The thickness, the samples being from 1.42 to 3.20 mm thick, that ordinarily used in spectacle lenses.
5. The per cent transmission curve from 230 mμ to 710 mμ, in each figure being given for comparison the transmission curve of a sample of colorless crown glass and the visibility curve for the average human eye,
6. The total transmission factor for light of equal energy at all wave lengths.

Four different methods were used to obtain the data, part of the work being done in the physical laboratory of Cornell University and part at this Bureau.

Sample transmission curves are given with all values plotted, illustrating the kind of agreements and disagreements obtained on the different instruments.

A brief summary is given of the good or bad qualities of the various kinds of glass in comparison with colorless glass as regards protection against ultra-violet radiant energy.

A brief elementary discussion is given, explaining the meaning of terms used in spectrophotometry and illustrating how to compute the transmissions for thicknesses other than that measured.

APPENDIX

FORMS SUITABLE TO BE USED IN THE SPECIFICATIONS OF EYE-PROTECTIVE GLASSES

It is desirable that those who wish to specify eye-protective glasses should have at hand certain standard type forms for such specifications. The following forms, separately or in combination, as the case in hand may require, will be found, in general, suitable and adequate for this purpose:

1. *For Protection from Ultra-Violet.*—"The transmission shall not be greater than 0.01 for radiant energy of any wave length less than 406 millimicrons."

2. *For Protection from Heat.*—"The total transmission for radiant energy from a source having the spectral energy distribution of a complete radiator ('black body,' Wien equation) at 1500° absolute shall not be greater than ....."

3. *For Protection from Intense Light while Allowing Sufficient Light for Work.*—"The total light transmission for light of equal energy at all wave lengths shall not be more than ..... nor less than ....." This would approximate closely the total light transmission for sunlight. In some cases it might be desirable to make the specification in terms of the known spectral energy distribution of the standard acetylene flame or other source.

The blank spaces in the above forms should be filled as found desirable in particular cases. Satisfactory glasses should be selected by trial under service conditions. The measured transmissions of these selected glasses will then serve as data for filling the blanks in the above forms.

WASHINGTON, July 25, 1918.

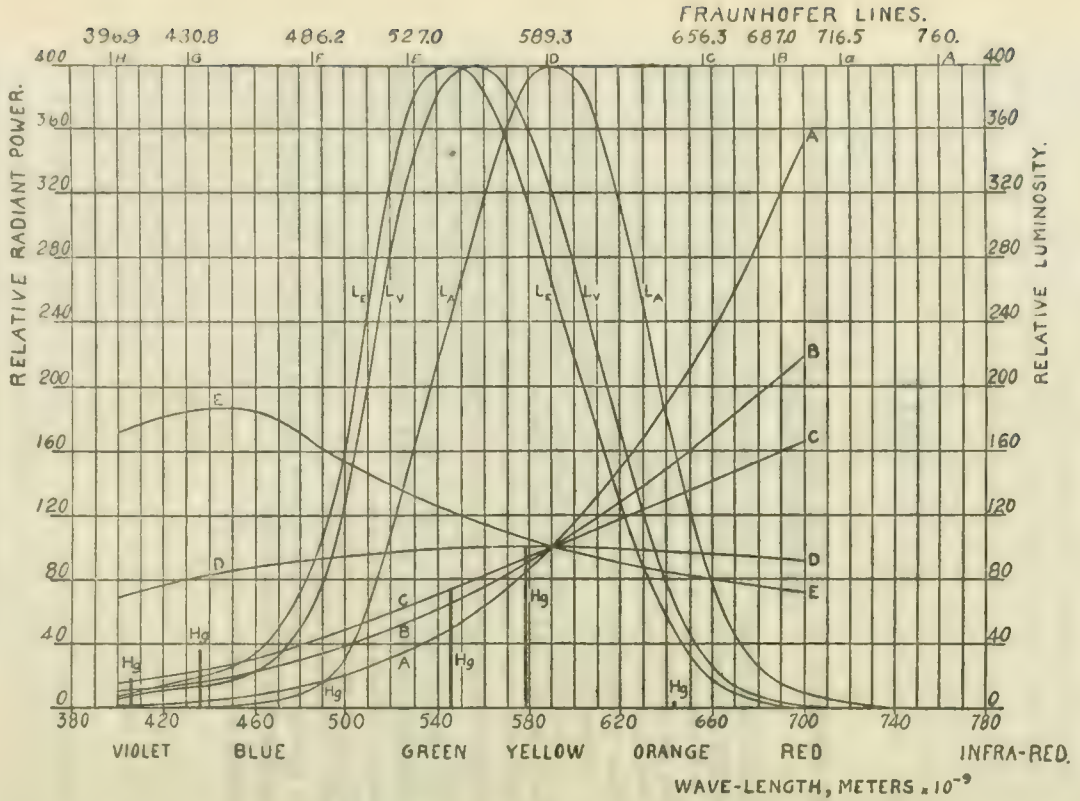


FIG. 1

Relative spectral distribution of radiant power in various sources: *A*=Hefner lamp, H. E. Ives, Trans. I. E. S., 5, p. 208, 1910; *B*=acetylene flame, Coblentz and Emerson, B. S., 13, p. 363, 1916; *C*=tungsten (gas) incandescent lamp (No. 1717) at 15.6 lumens per watt, data by Coblentz; *D*=black body at 5,000° absolute—approximately sunlight, computed from Planck equation; *E*=blue sky, H. E. Ives, Trans. I. E. S., 5, p. 208, 1910; *Hg*=Heraeus quartz-mercury lamp, 81 watts, W. W. Coblentz, B. S., 9, p. 97, 1913.

Relative visibility: L_V=Relative visibility curve for the average human eye (or luminosity of a source having constant radiant power throughout the visible spectrum), Coblentz and Emerson, B. S., 14, p. 192, 1917.

Relative luminosity=relative radiant power times relative visibility: L_A=luminosity of Hefner lamp; L_B=luminosity of blue sky.

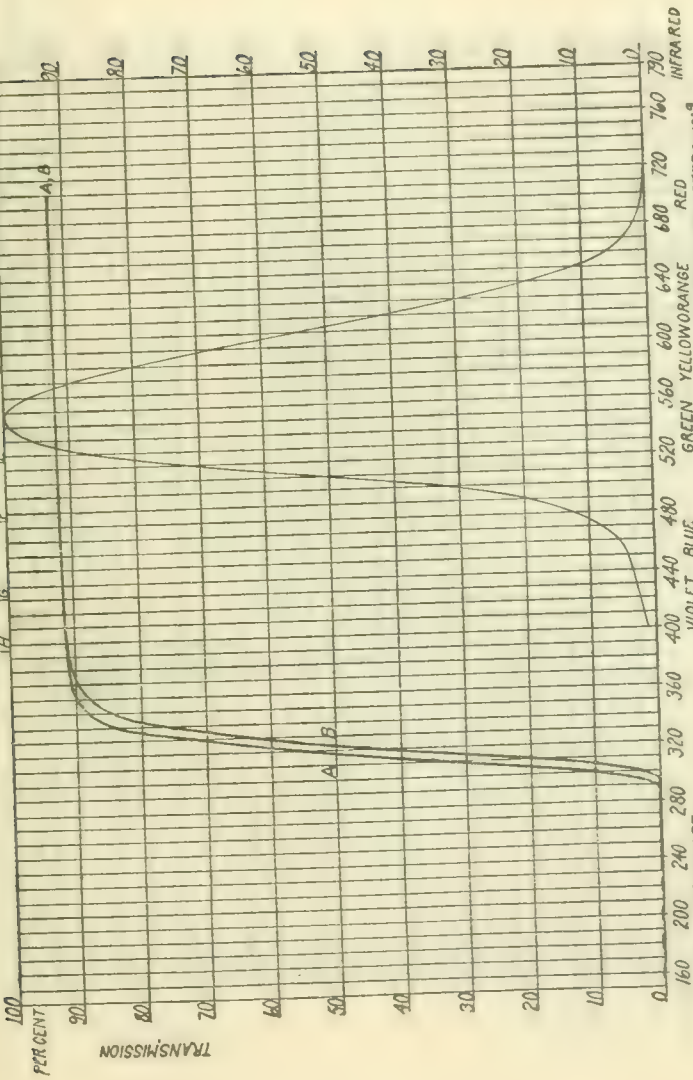

FRAUNHOFER LINES
3969 4308 4862 5270 5893 6563 687 7165 760
H K F E D C B A
100 90 80 70 60 50 40 30 20 10 0
PERCENT TRANSMISSION
160 200 240 280 320 360 400 440 480 520 560 600 640 680 720 760 790
ULTRA-VIOLET VIOLET BLUE GREEN YELLOW ORANGE RED INFRA RED
WAVE-LENGTH, METERS ×10⁻⁹
A
B
A, B

FIG. 2.—A = "Crown 1.50," 1.68 *mm*; B = "1.52," 2.33 *mm*; A. O. Co.

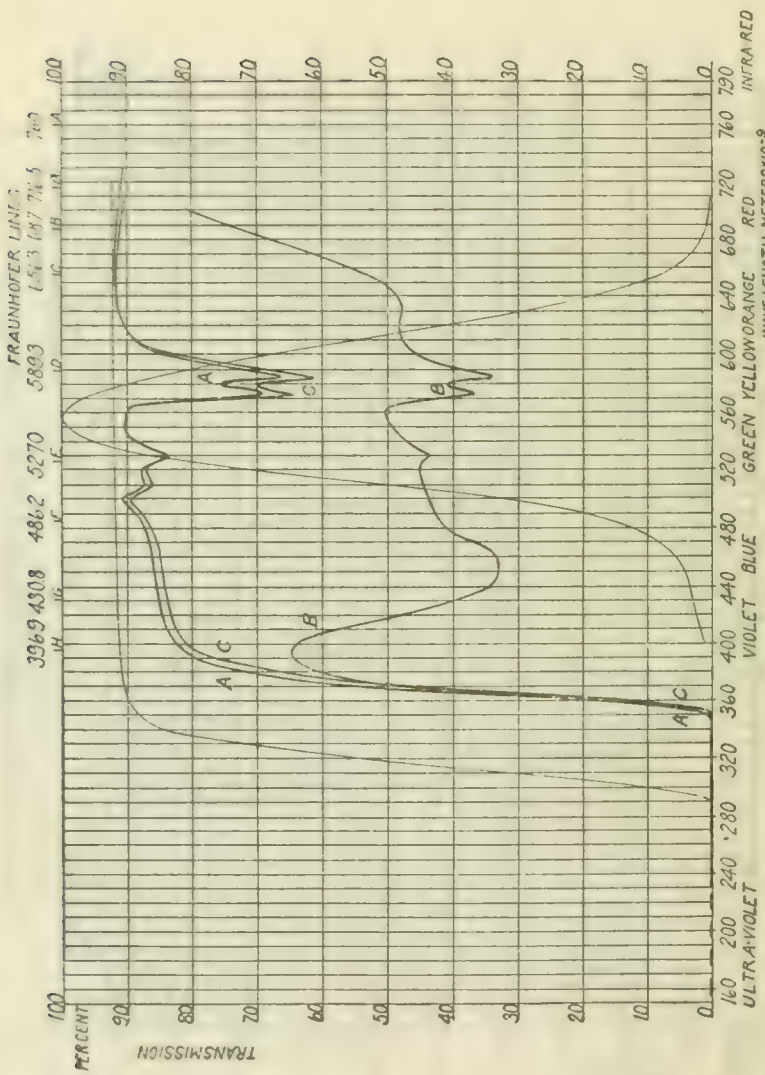


FIG. 3.—*A* = "Crookes A, Wellsworth," 2.05 mm; *B* = "Crookes B, Wellsworth," 2.16 mm; A. O. Co. *C* = "91 B," 1.97 mm; Corning

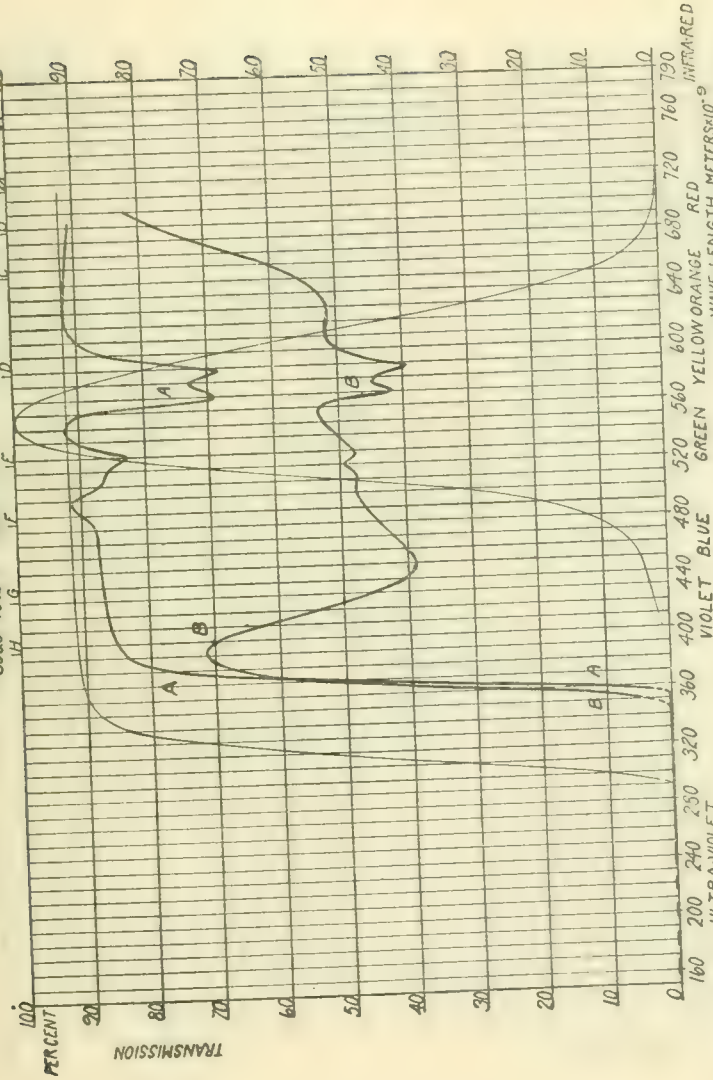


FIG. 4.—A = "Crookes A," 1.79 mm; B = "Crookes B," 1.93 mm; W. & O.

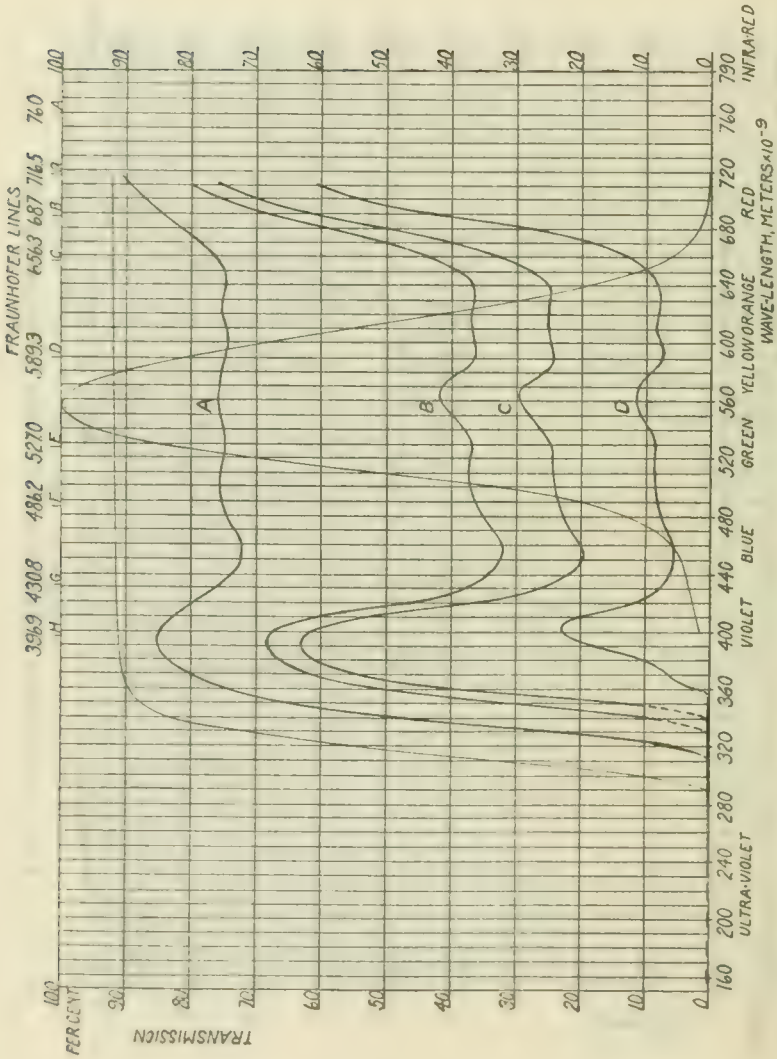


FIG. 5.—*A* = "*Smoke A*," 2.10 *mm*; *B* = "*Smoke B*," 2.14 *mm*; *C* = "*Smoke C*," 2.13 *mm*; *D* = *Smoke D*," 2.03 *mm*; *A. C. Co.*

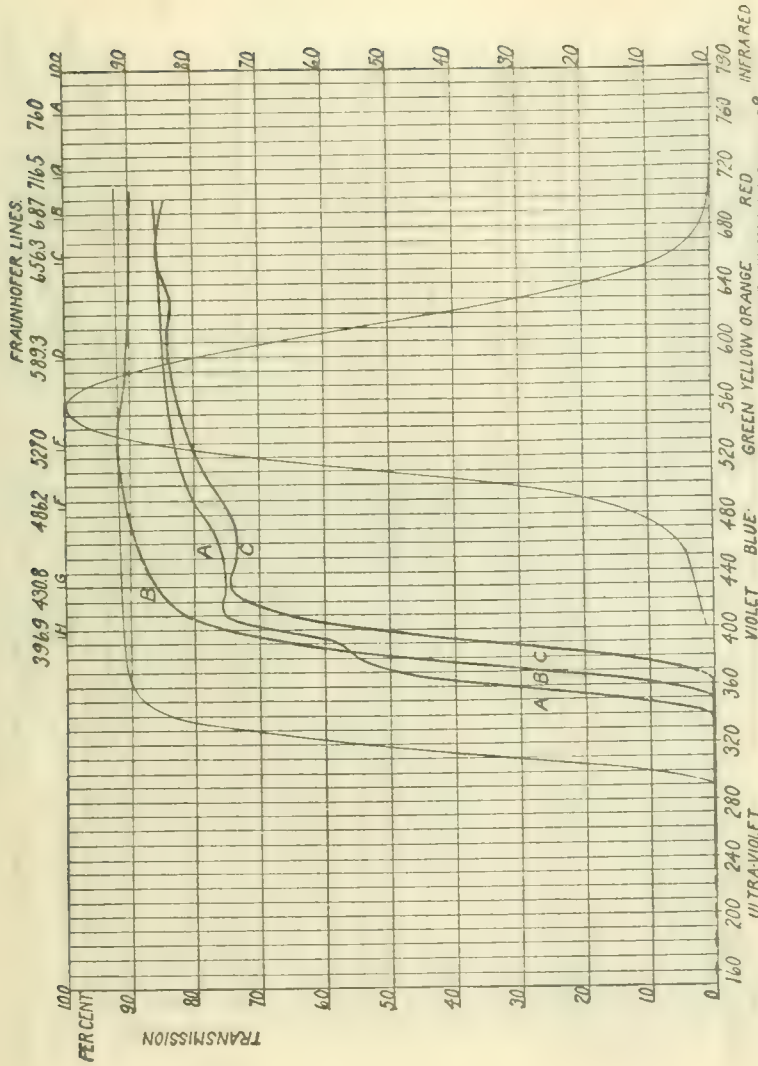


FIG. 6.—*A* = "Luxfel," 2.00 *mm*; *B* = "Lab. No. 57," 1.90 *mm*; *C* = "Lab. No. 58," 2.02 *mm*; *A. O. Co.*

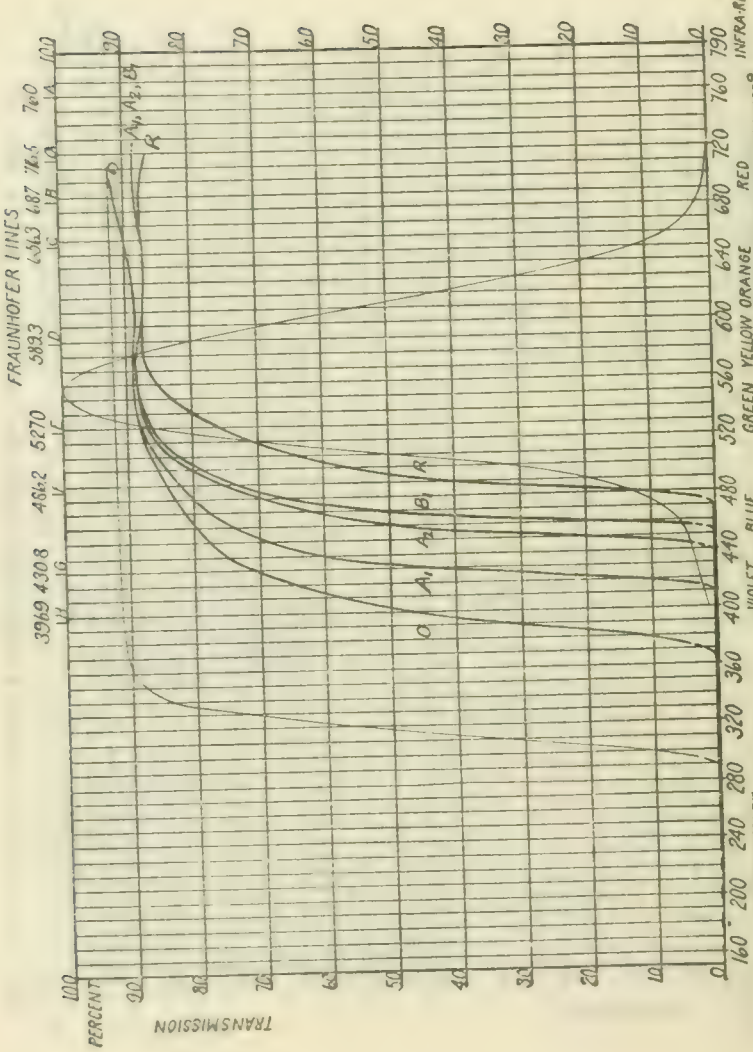


FIG. 7.—O="Noviol O," 1.04 mm; Hardy. A_1="Noviol A_1," 2.88 mm; A_2="Noviol A_2," 2.84 mm; B_1="Noviol B_1," 2.83 mm; Corning. R="Rifleite," 3.14 mm; King

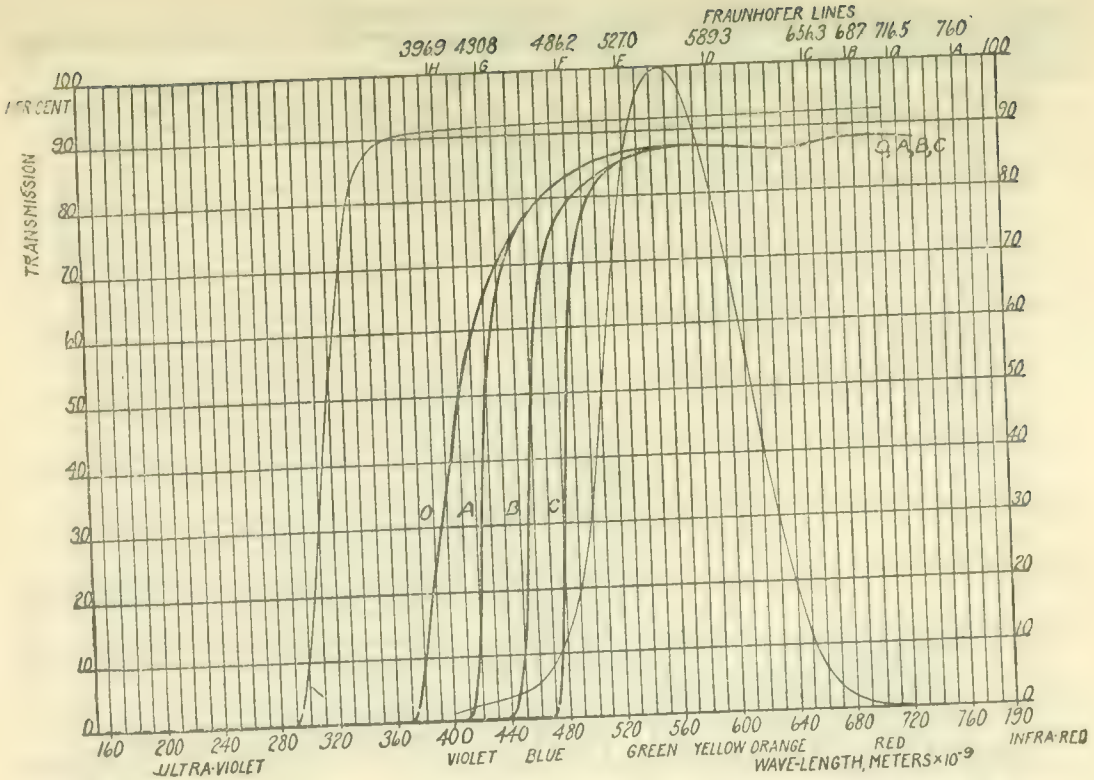


FIG. 8.—*O*="*Noviol O*," 2.01 *mm*; *A*="*Noviol A*," 2.00 *mm*; *B*="*Noviol B*," 1.95 *mm*; *C*="*Noviol C*," 2.00 *mm*; *Corning*

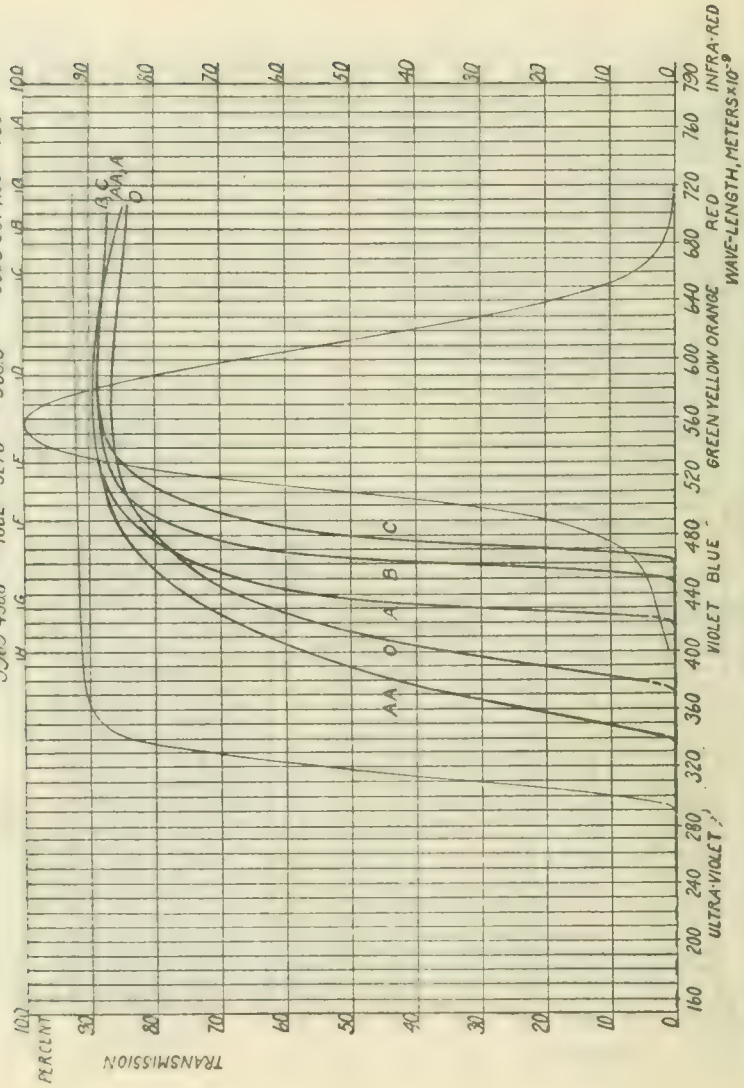


FIG. 9.—*AA* = "Noviol *AA*," 1.88 mm; *O* = "Noviol *O*," 2.03 mm; *A* = "Noviol *A*," 2.06 mm; *B* = "Noviol *B*," 2.10 mm; *C* = "Noviol *C*," 2.10 mm; A. O. Co.

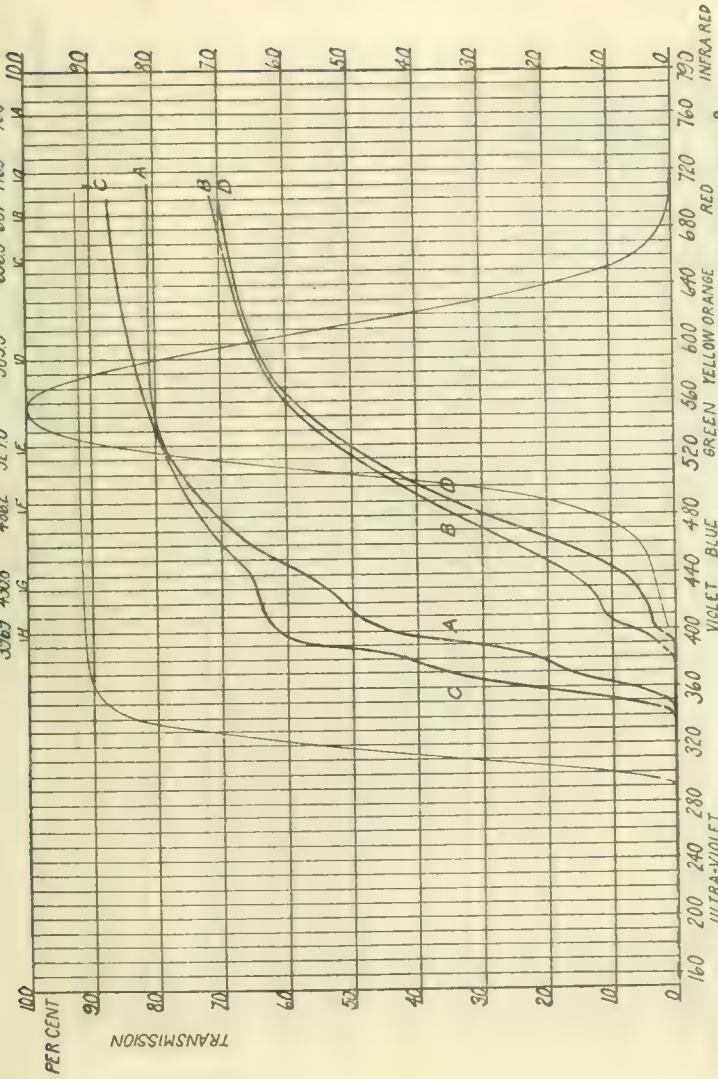


FIG. 10.—*A*="*Amber A*," 2.13 *mm*; *B*="*Amber B*," 2.13 *mm*; *A. O. Co. C*="*Amber, light*," 1.93 *mm*; *D*="*Amber, dark*," 1.85 *mm*; *W. & O.*

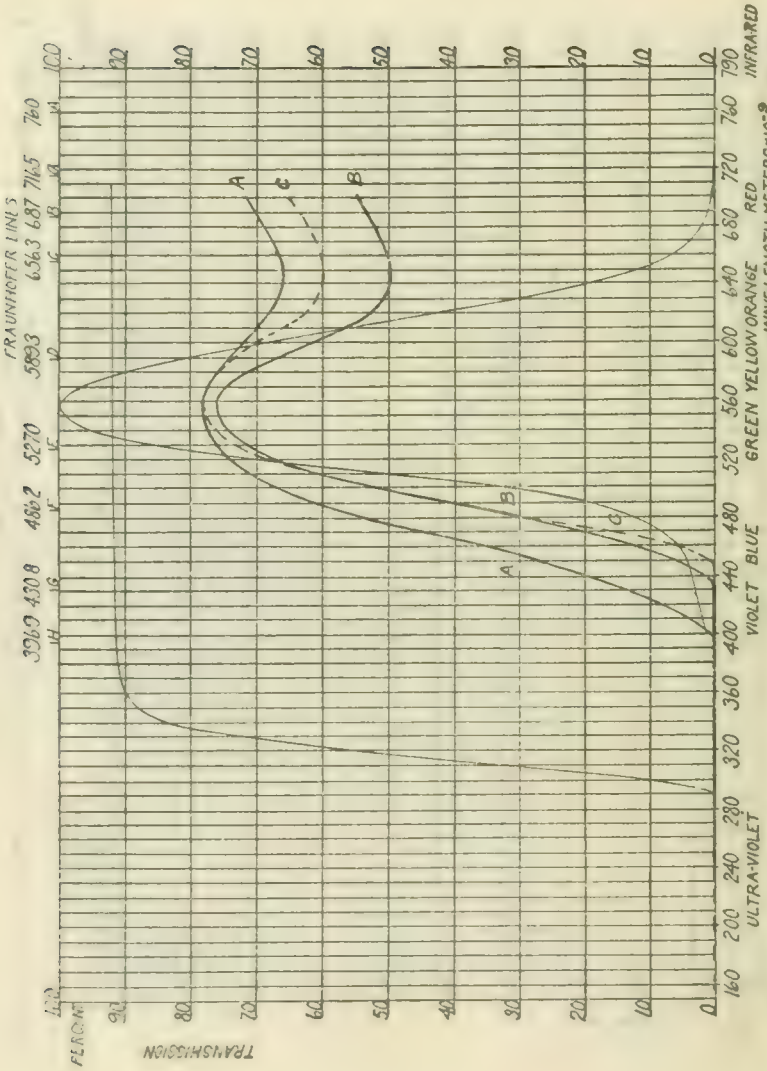


FIG. 11.—A = "Euphos," 1.95 mm; B = "Lab. No. 61," 2.13 mm; A. O. Co. C = glass labeled "Fieuzal," bought in store

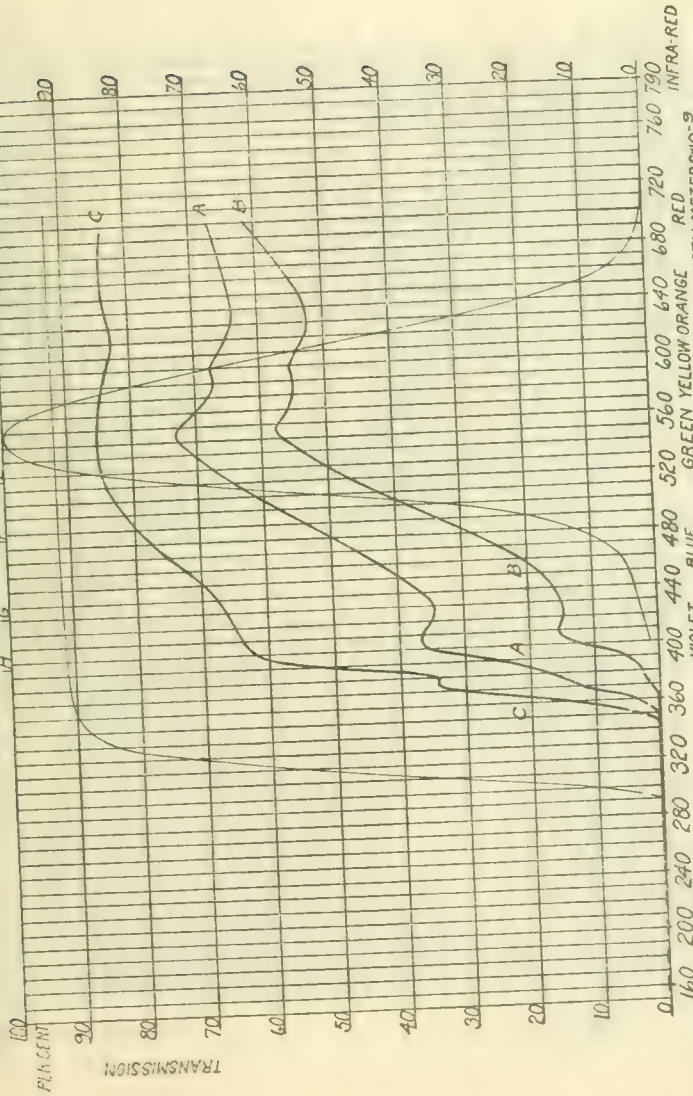


FIG. 12.—A = "Fieuzal A," 2.13 mm; B = "Fieuzal B," 2.13 mm; A. O. Co. C = "Fieuzal," 1.98 mm, W. & O.

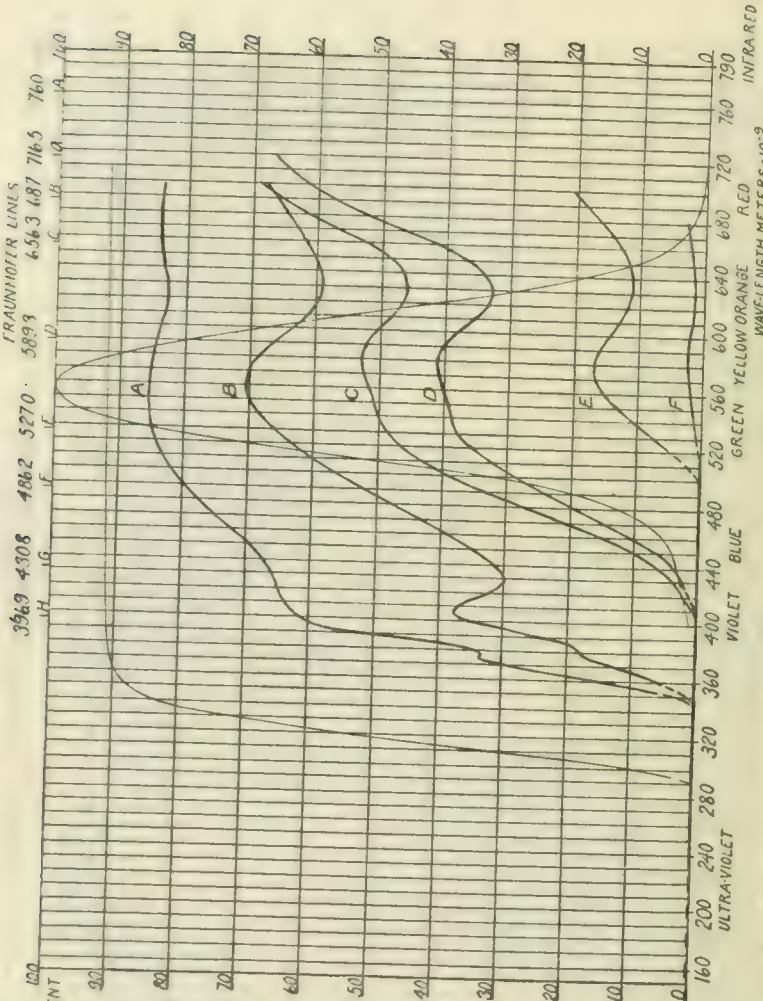


FIG. 13.—*A*="*Fiunal*," 1.98 *mm*; *B*="*Chlorophile*," 1.98 *mm*; *C*="*Hallauer*," 1.90 *mm*; *W. & O. D*="*Ahopos*," 2.17 *mm*; *E*="*Saniweld, light*," 1.82 *mm*; *F*="*Saniweld, dark*," 2.12 *mm* (*see Fig. 21*); *King*

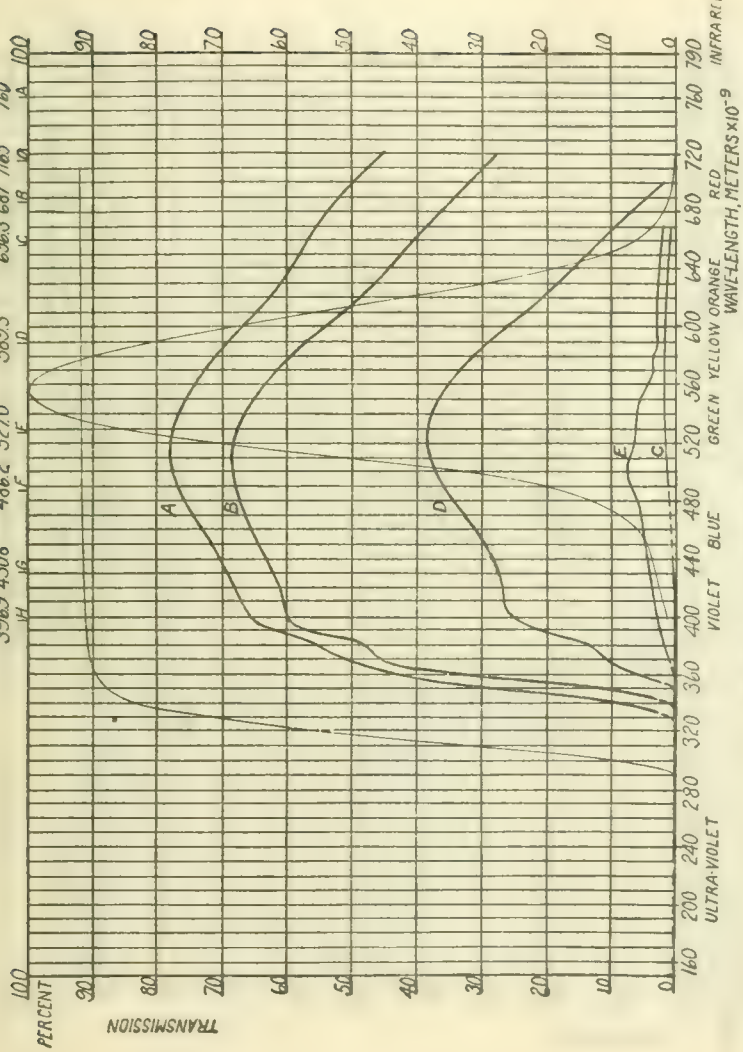

FRAUNHOFER LINES
3963 4308 486.2 5270 5893 6563 687 7165
H K F E D C B A
PERCENT
TRANSMISSION
100 90 80 70 60 50 40 30 20 10 0
160 200 240 280 320 360 400 440 480 520 560 600 640 680 720 760 790
ULTRA-VIOLET VIOLET BLUE GREEN YELLOW ORANGE RED INFRARED
WAVE-LENGTH, METERS x10⁻⁹
A
B
C
D
E

FIG. 14.—*A*="392 F," 1.00 *mm*; *B*="124 JA," 2.02 *mm*; *C*="124 IP," 2.00 *mm* (see Fig. 21); *Corning*. *D*="Lab. No. 59," 2.13 *mm*; *E*—"Pfund," gold film between plates of "Crookes" glass, total 2.89 *mm*; *A. O. Co.*

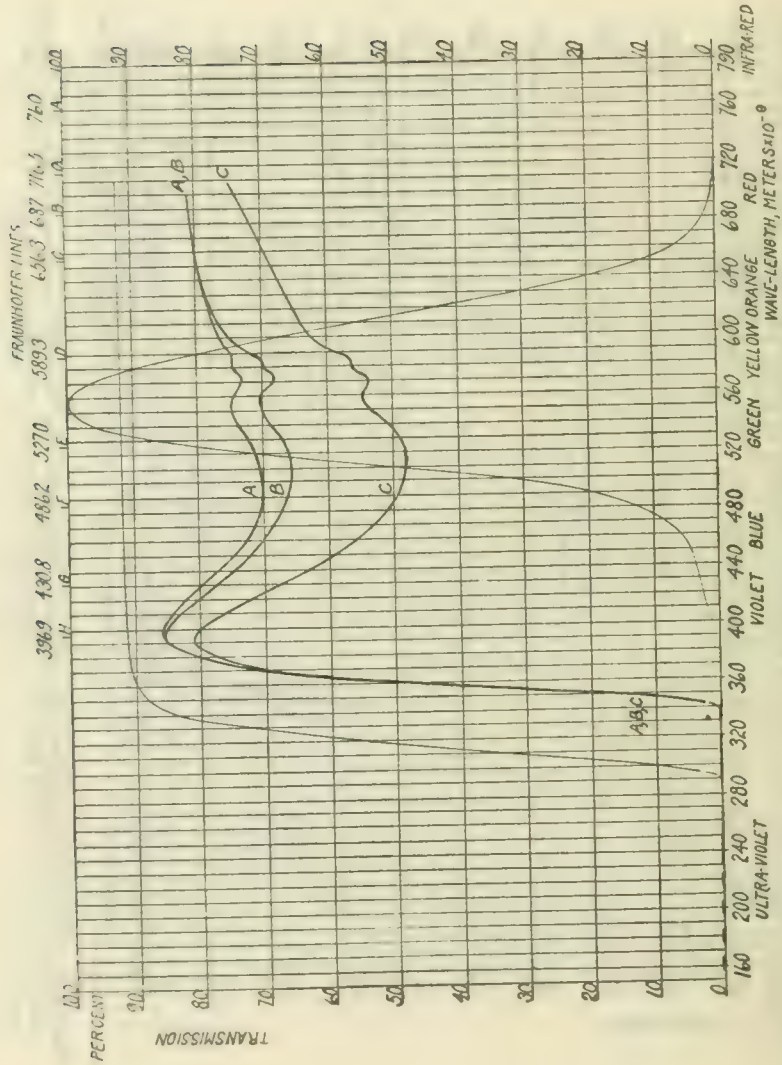


FIG. 15.—A = "Amethyst A," 2.08 mm; B = "Amethyst B," 2.05 mm; C = "Amethyst C," 2.04 mm; A. O. Co.

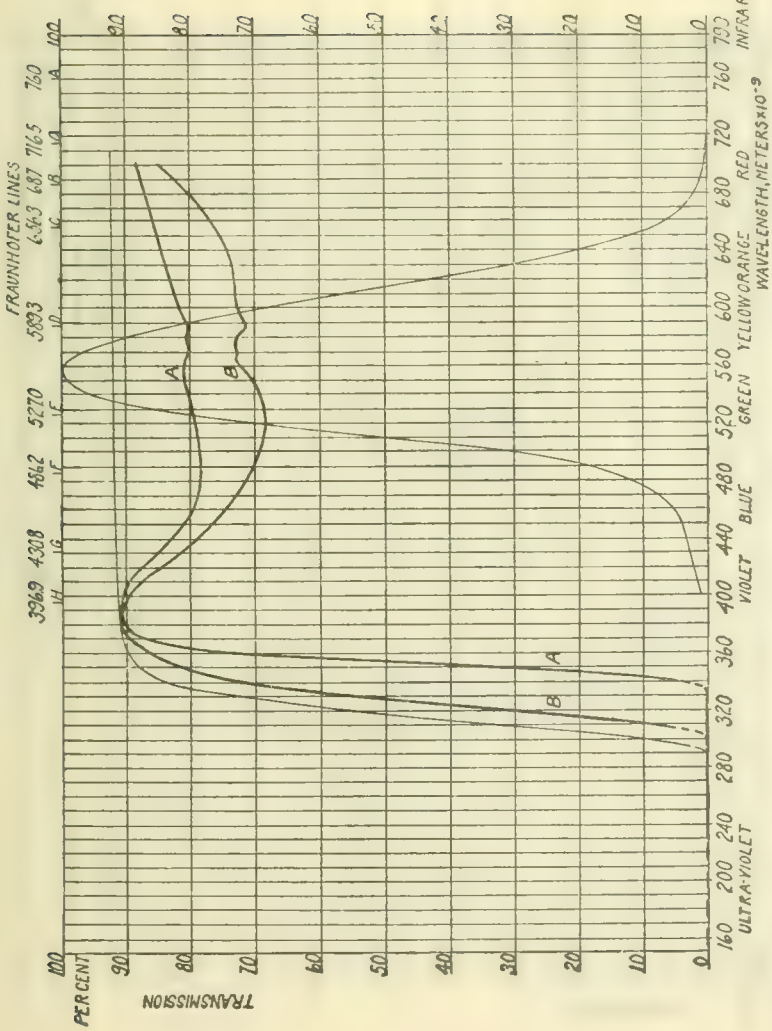


FIG. 16.—*A* = "*Amethyst, light,*" *1.97 mm; B* = "*Amethyst, dark,*" *1.90 mm; W. & O.*

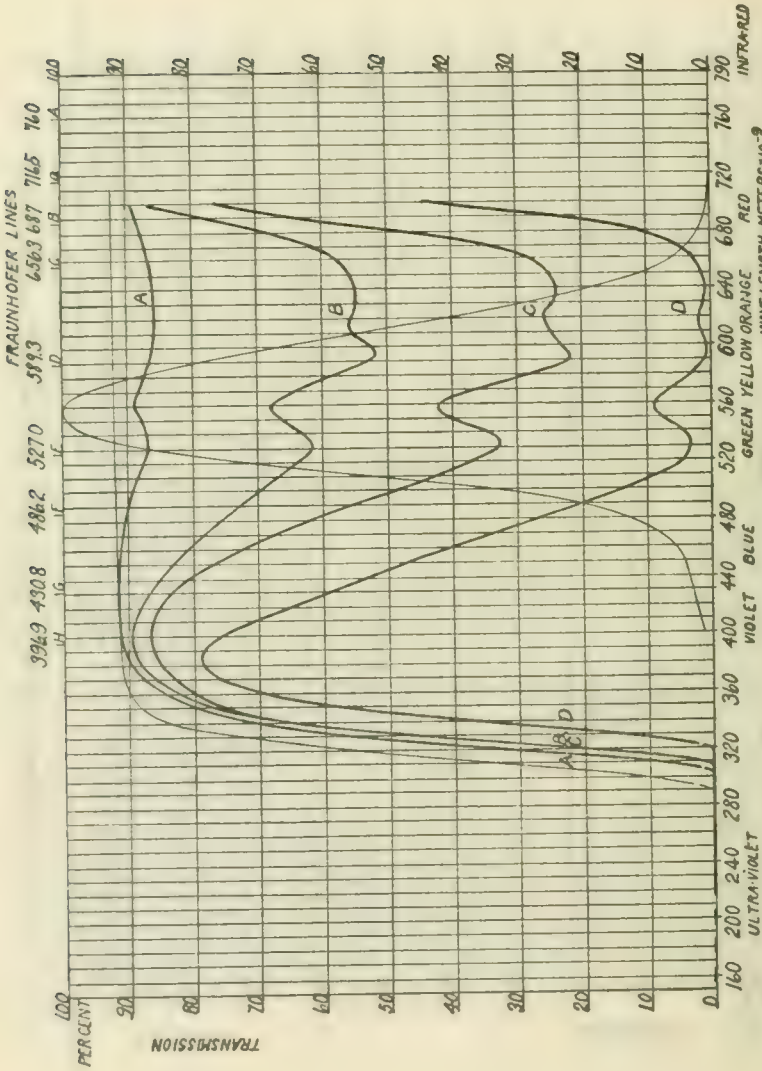

FRAUNHOFER LINES
3249 4308 4862 5270 5893 6563 687 7165 760
H δ F E D C B A
TRANSMISSION
PER CENT
100 90 80 70 60 50 40 30 20 10 0
160 200 240 280 320 360 400 440 480 520 560 600 640 680 720 760 790
ULTRA-VIOLET VIOLET BLUE GREEN YELLOW ORANGE RED INFRA-RED
WAVE-LENGTH, METERS×10⁻⁹
A
B
C
D

FIG. 17.—A = "Blue A," 2.10 mm; B = "Blue B," 2.04 mm; C = "Blue C," 2.05 mm; D = "Blue D," 2.11 mm; A. O. Co.

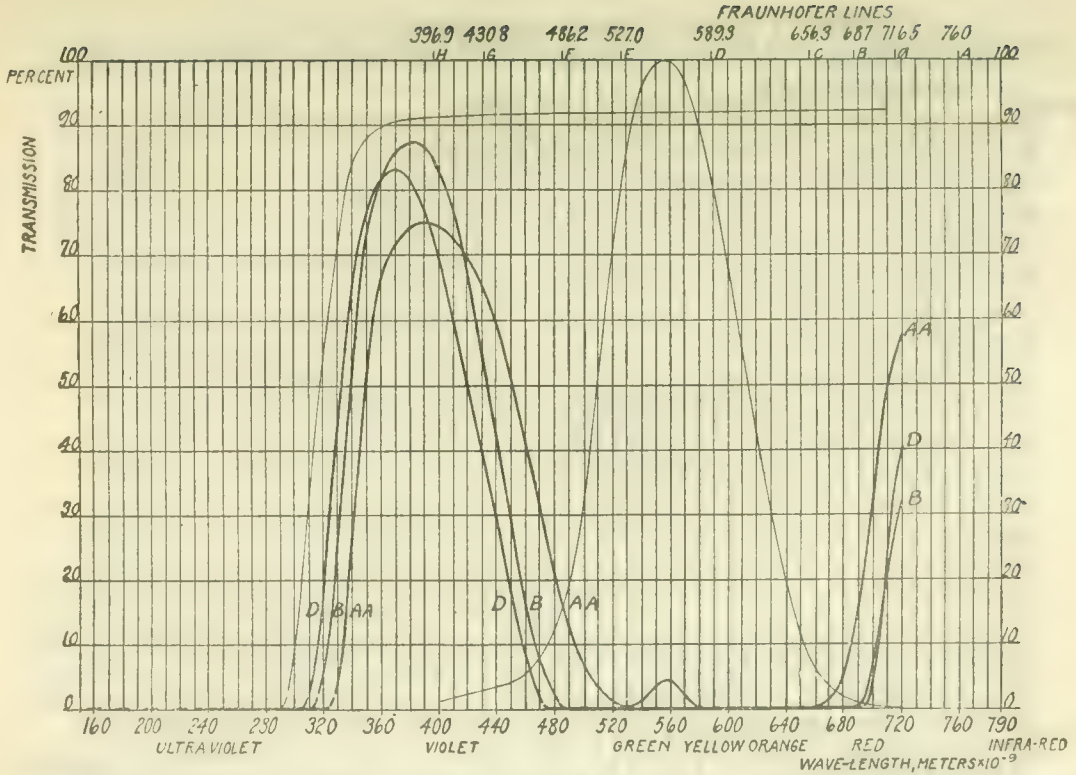


FIG. 18.—*AA*="*Cobalt Blue AA*," *2.75 mm; B*="*Cobalt Blue B*," *1.85 mm; D*="*Cobalt Blue D*," *1.86 mm; "Cobalt Blue A," 3.20 mm nearly same as curve AA; "Cobalt Blue C," 1.46 mm nearly same as curve B; "Chromatic Test," 2.36 mm similar to curve B, but slightly lower in value; A. O. Co.*

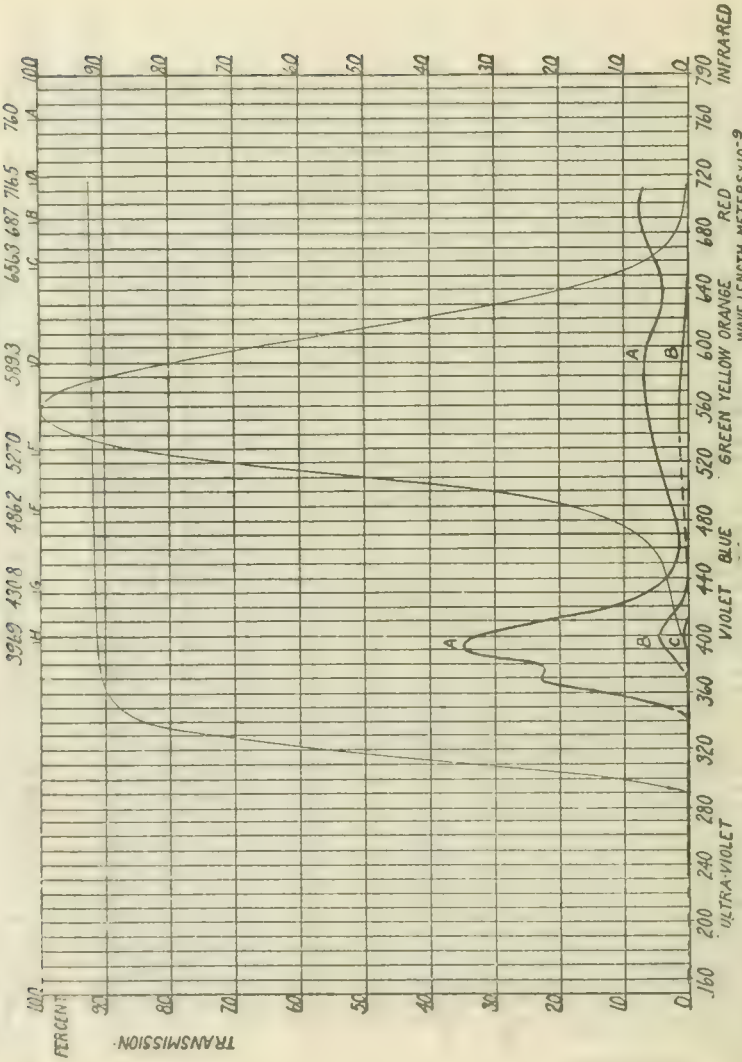


FIG. 19.—A = "Welders Smoke Dark," 1.42 mm; B = "Special Welders Light," 1.68 mm (see Fig. 21); C = "Special Welders Dark," 2.54 mm (see Fig. 21); A. O. Co.

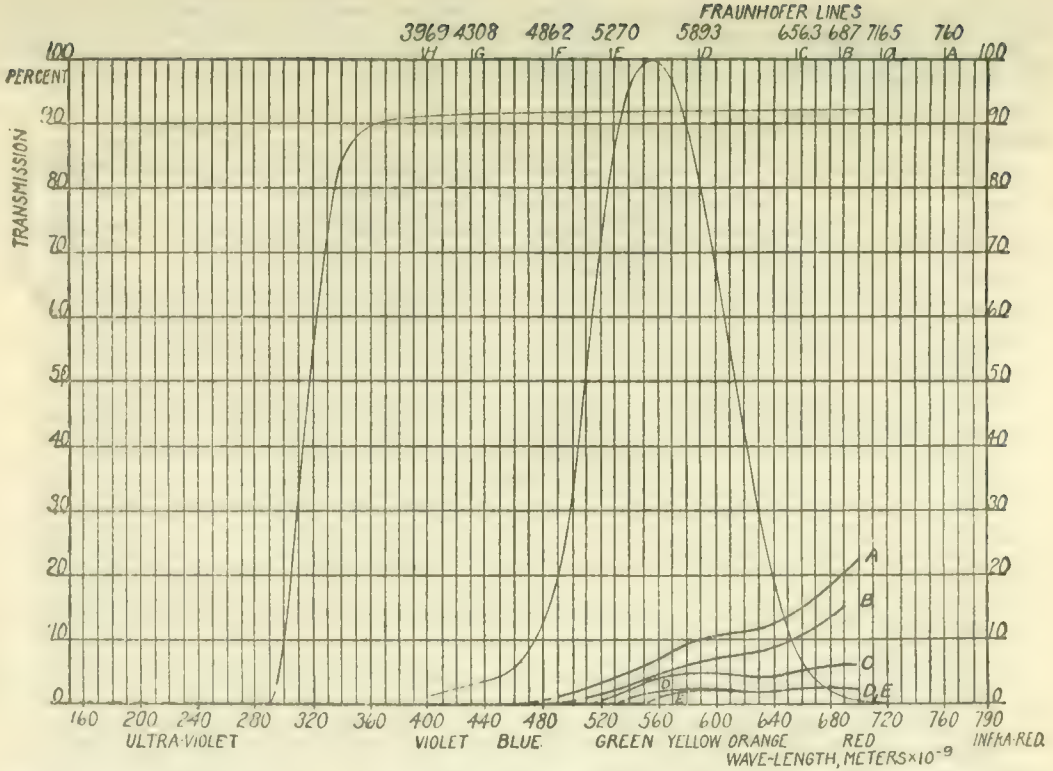


FIG. 20 — A = "Welding glass 6," 1.97 mm; B = "Welding glass 2," 2.32 mm; C. E. S. Co. C = "Noviweld 4," 1.89 mm; D = "Noviweld 5," 2.16 mm; E = "Noviweld 6," 2.20 mm; "Noviweld 7," 1.90 mm 1.02% at 578; "Noviweld 8," 2.01 mm 0.517% at 578; A. O. Co.

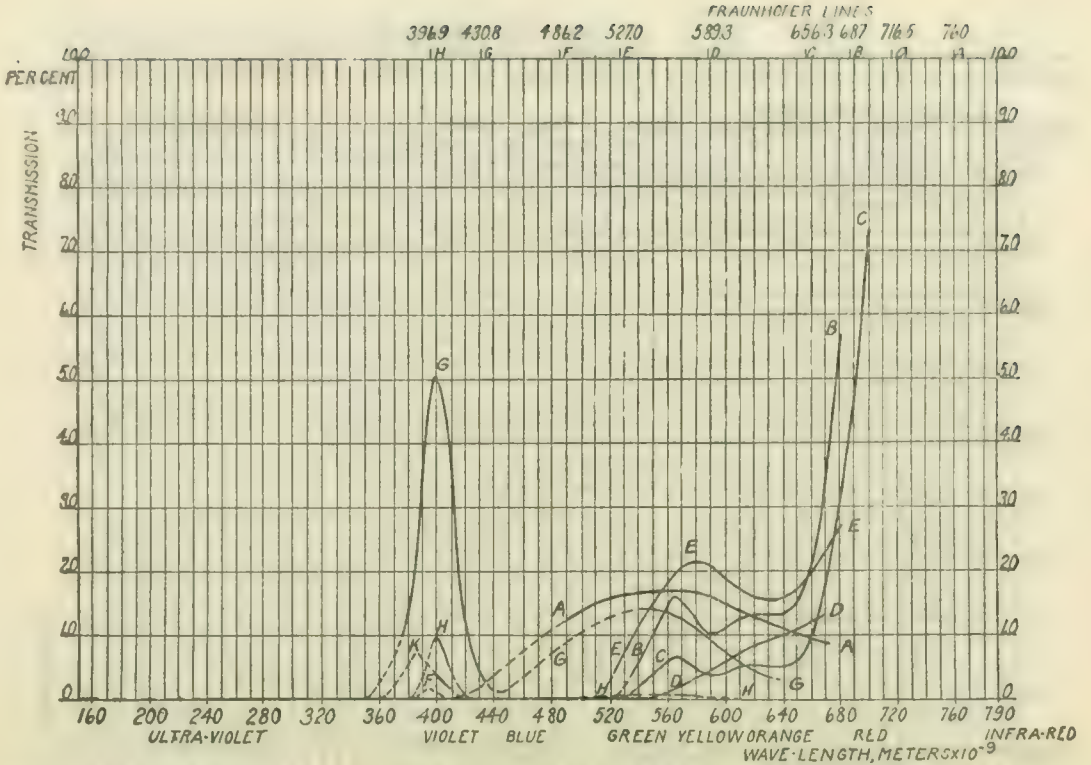


FIG. 21.—*Note scale of ordinates. A="124 IP," 2.00 mm (see Fig. 14); Corning. B="Welding glass 3," and "Welding glass 4," 1.48 mm (identical); C="Welding glass 1," 1.98 mm; D="Welding glass 5," 1.87 mm C. E. S. Co. E="Saniweld, Dark," 2.12 mm (see Fig. 13); F="Ankweld, Dark," 2.46 mm; King. G="Special Welders Light," 1.68 mm (see Fig. 19); H="Special Welders Dark," 2.54 mm, 0.001% at 578 (see Fig. 19); K="Electric Smoke Red," 1.82 mm; A. O. Co.*

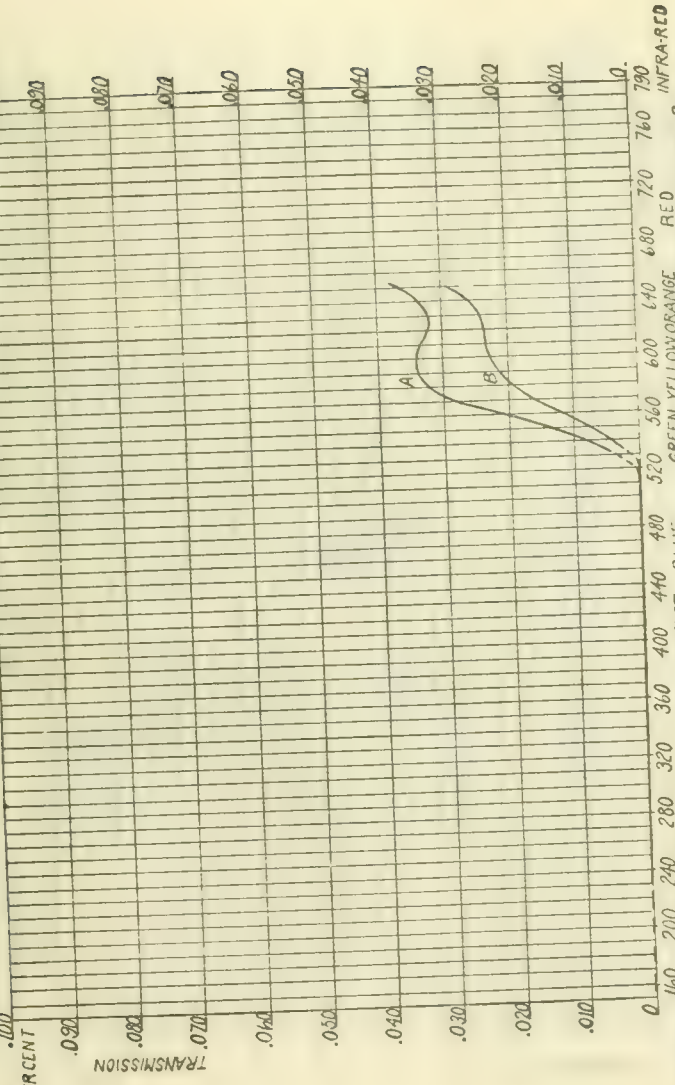


FIG. 22.—*Note scale of ordinates.* *A* = "*Special Noviweld No. 8*," *1.77 mm; Hardy*. *B* = "*391 DD*," *1.90 mm; Corning.*

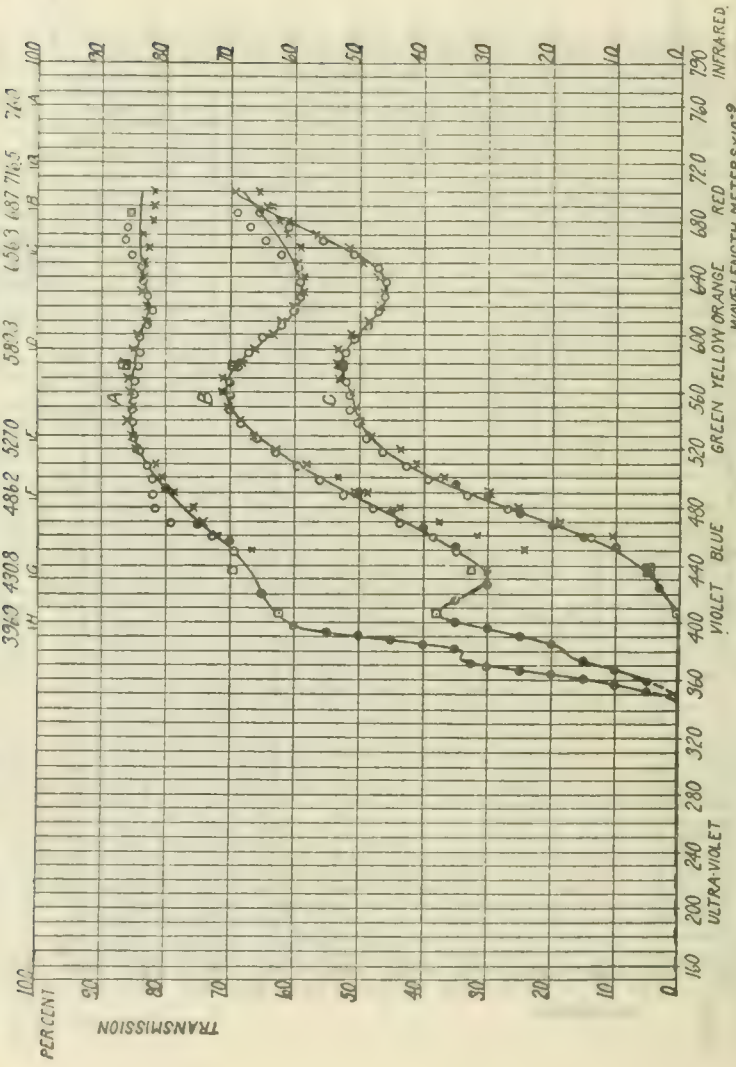


FIG. 23.—A="Fieuzal," 1.98 mm; B="Chlorophile," 1.98 mm; C="Hallauer," 1.90 mm (see Fig. 13); W. & O.

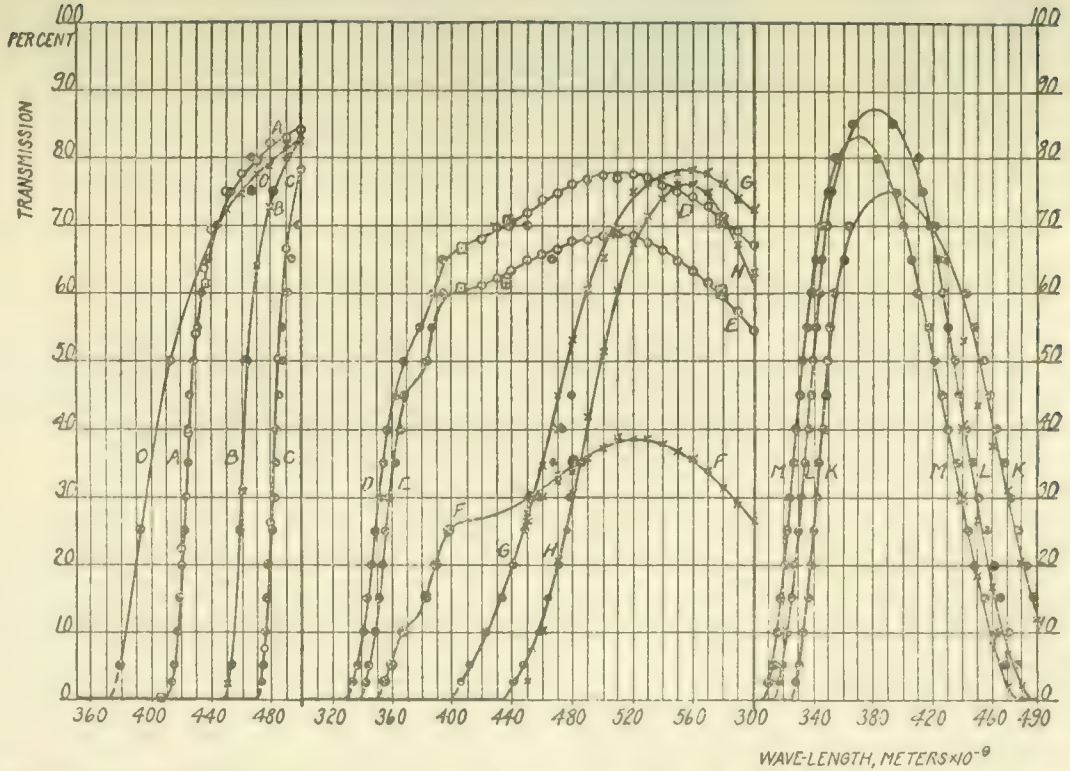


FIG. 24.—O="Noviol O," 2.03 mm; B="Noviol B," 2.10 mm (see Fig. 9); A. O. Co. A="Noviol A," 2.00 mm; C="Noviol C," 2.00 mm (see Fig. 8); Corning. D="392 F," 1.90 mm; E="124 JA," 2.02 mm (see Fig. 14); Corning. F="Lab. No. 59," 2.13 mm (see Fig. 14); G="Euphos," 1.95 mm; H="Lab. No. 61," 2.13 mm (see Fig. 11); A. O. Co. K="Cobalt Blue AA," 2.75 mm; L="Cobalt Blue B," 1.85 mm; M—"Cobalt Blue D," 1.86 mm (see Fig. 18); A. O. Co.

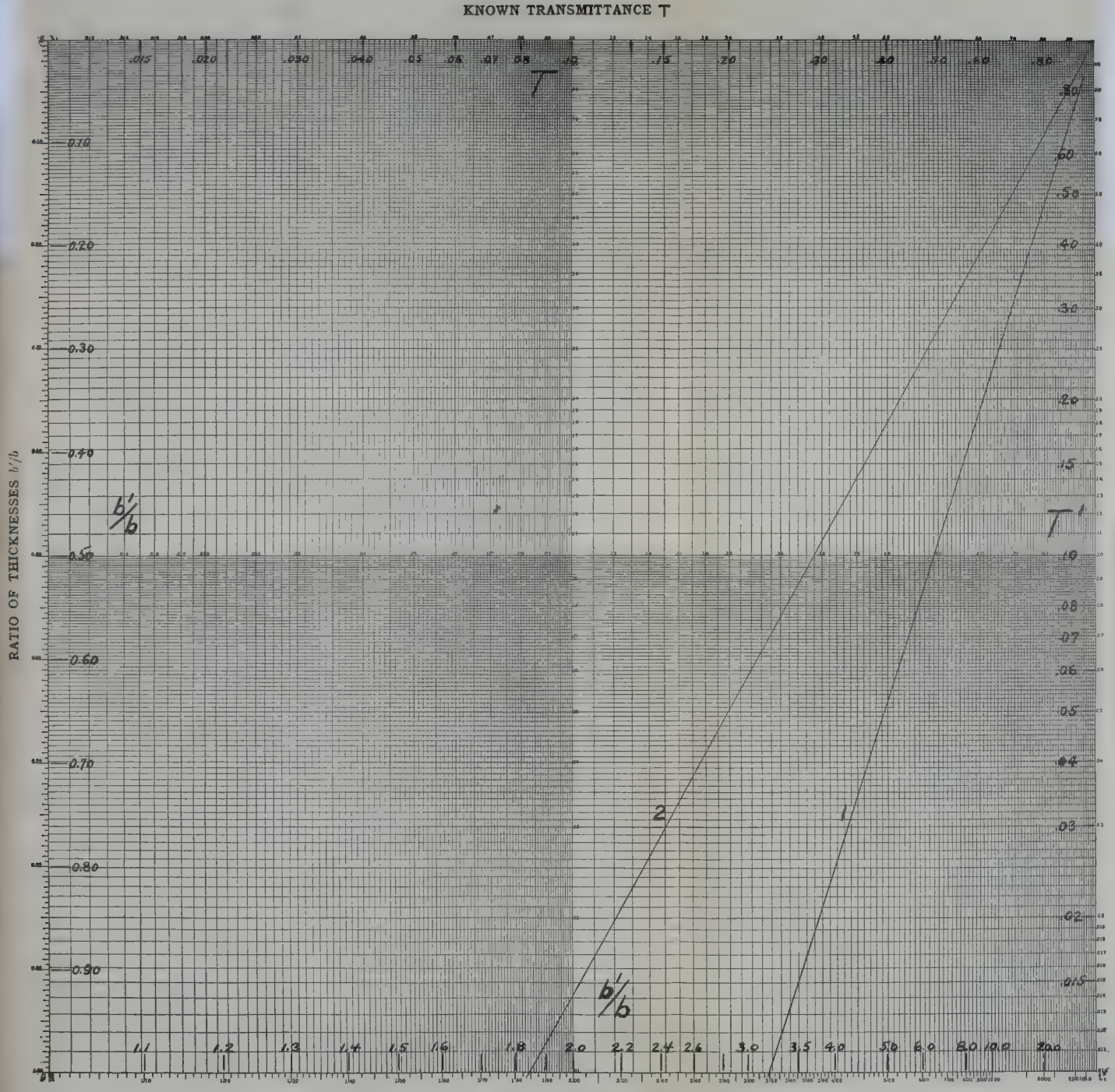


FIG. 25.—*Reproduction of chart used at Bureau of Standards for computing transmissions for different thicknesses and vice versa*

Method of use.—To find T' when b', T, and b are given: Lay a straight edge from origin in upper right-hand corner to value of b'/b found on left-hand or lower scale. Find τ ($=T/0.92$) on upper scale and follow it down to intersection with the straight edge. The value of τ' on right-hand scale corresponding to this intersection is the required transmittance and $T'=0.92\ \tau'$. Line 1 illustrates the solution of problem 3 in Table 1.

To find b' when T', T, and b are known Lay a straight edge from upper right-hand corner through the intersection of τ and τ' ($=T'/0.92$ and $T'/0.92$, respectively). The intersection of this straight edge on the lower scale gives b'/b, from which b' can be found. Line 2 illustrates the solution of problem 5 in Table 1.

89784°—19. (To follow page 47.)

DEPARTMENT OF COMMERCE

TECHNOLOGIC PAPERS

OF THE

BUREAU OF STANDARDS

S. W. STRATTON, DIRECTOR

No. 120

TESTS OF HOLLOW BUILDING TILES

BY

BERNARD D. HATHCOCK, Laboratory Assistant

and

EDWARD SKILLMAN, Laboratory Assistant

Bureau of Standards

ISSUED FEBRUARY 8, 1919

PRICE, 5 CENTS

Sold only by the Superintendent of Documents, Government Printing Office
Washington, D. C.

WASHINGTON
GOVERNMENT PRINTING OFFICE
1918

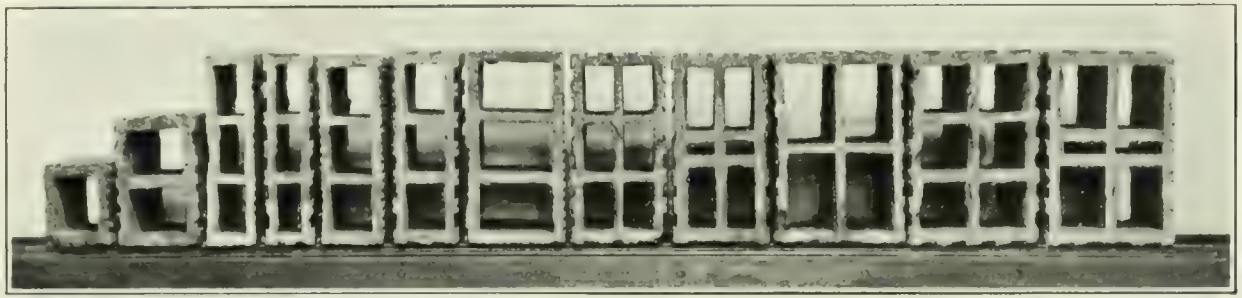


FIG. 1.—*Tiles (from left to right) 4 by 5 by 12 inches; 5 by 8 by 12 inches, 3 by 12 by 12 inches, type 1; 3 by 12 by 12 inches, type 2; 4 by 12 by 12 inches, type 1; 4 by 12 by 12 inches, type 2; 6 by 12 by 12 inches, type 1; 6 by 12 by 12 inches, type 2; 6 by 12 by 12 inches, type 3; 8 by 12 by 12 inches, type 1; 8 by 12 by 12 inches, type 2; 8 by 12 by 12 inches, type 3.*

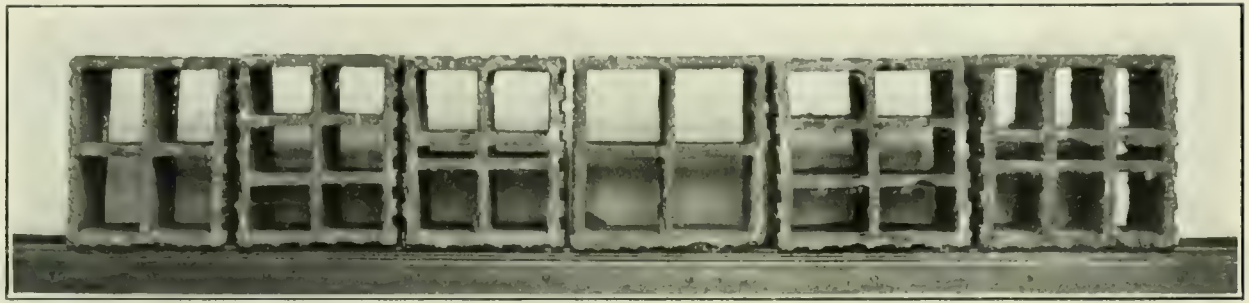


FIG. 2.—*Tiles (from left to right) 10 by 12 by 12 inches, type 1; 10 by 12 by 12 inches, type 2; 10 by 12 by 12 inches, type 3; 12 by 12 by 12 inches, type 1; 12 by 12 by 12 inches, type 2; 12 by 12 by 12 inches, type 3.*

TESTS OF HOLLOW BUILDING TILES

By Bernard D. Hathcock and Edward Skillman

CONTENTS

	Page
I. Introduction	3
II. Scope of tests	5
III. General procedure of tests	6
1. Measuring and weighing of tiles	6
2. Compression tests	6
IV. Details of compression tests	6
V. Absorption tests	7
VI. Results and discussion of tests	8
1. Weight per cubic foot	8
2. Compression tests	8
3. Absorption tests	17
VII. General summary of tests	19

I. INTRODUCTION

Hollow building tile has attained a place of considerable distinction as a structural material, and is used to-day for a variety of purposes, although it was comparatively unknown a few decades ago. Originally it was used mostly for its fire-resisting properties, but, as its other advantages have become more generally recognized, its range of usefulness has been broadened to a great extent. The following are some of its principal advantages: (1) It requires comparatively less labor in construction, as the space occupied by one tile is quite large; (2) it is relatively impervious to moisture; (3) it possesses good heat-insulating qualities; (4) it effects a saving in clay compared to solid brick because of the hollow cells of the tiles and a consequent reduction in the cost of transportation; (5) it possesses good fire-resisting properties; (6) it is a poor conductor of sound; and (7) it has sufficient strength to meet the needs of building practice under the limitations of adequate specifications. Of these several advantages this paper is limited to a discussion of the one pertaining to the strength of the tiles.

Tiles are made of red and buff-burning clays and sometimes fire clay possessing sufficient plasticity for molding into shapes and sizes according to their particular use. Sufficient water is added to the clay to insure a plastic state after kneading. Then it is

molded by being pressed through a die of the desired shape and size, dried by hot air in tunnel driers, and burned in some form of kiln, usually of the down-draft type. The temperature of the burning process is moderate, ordinarily between 1900 and 2300° F, which is well beyond the stage of initial vitrification of the clay but rarely high enough to complete vitrification. Tiles are made with several degrees of porosity which are produced by burning to different temperatures or sometimes by mixing sawdust with the clay before molding. The sawdust burns out during the firing process and leaves small cavities throughout the structure. Very porous tile is light in weight and a splendid fire resistant, and is used only where strength is of minor consideration. Dense or only slightly porous tile is much heavier and stronger, and is generally used for exterior walls where strength is the principal determining factor.

The properties of the clays used and the degree of burning introduce factors which greatly influence the strength and the possible absorption of the tiles. Different manufacturers and even the same manufacturer at various times use clays which are more or less different in their characteristics; consequently the product is correspondingly varied as would naturally be expected. In a down-draft kiln the upper courses of tiles are heated to a higher temperature than the lower ones. This gives the top tiles a higher degree of burning, those near mid height a medium degree, and those near the bottom a lower degree of burning. In general, it may be said that the high-burned tiles are dark in color, the medium-burned of medium shade, and the low-burned light. However, this is not true in every case, for the natural color of some clays or the presence of coloring matter will cause a variation. In the case of red-burning clays the color may vary from a light red to a chocolate color.

Many tests have been made upon hollow building tiles from time to time, and the results obtained are valuable, although they have been insufficient for definite conclusions, partly because so few of the tests were upon tiles of any one kind and partly because no relations were established between their properties; however, previous tests were especially useful as a guide for this series, as they indicated in a general way what was to be expected and also formed a basis for the method of procedure.

The strength and other properties determined from samples of individual tiles are not always indicative of the properties of a

finished structure; nevertheless safe design of tile construction requires a thorough knowledge of the average strength of the individual tiles and the possible variations in order that allowances may be made by applying the proper factor of safety. Besides the need of tests from a designer's viewpoint, they are of great importance if not absolutely imperative in the formulation of building codes which pertain to tile construction. Of necessity these codes should be based only upon sound knowledge, as they represent the interest of the builder, the owner, and the public in general.

The tests collated in this paper were executed with such care and discretion as is ordinarily warranted in laboratory work of this nature, and the results obtained are as consistent as can reasonably be expected if due allowance is made for the numerous factors introduced by the materials used and the processes of manufacture which would naturally tend to vary the physical properties of the tiles.

The tiles used in these tests were manufactured in the State of Ohio and generously supplied to the Pittsburgh branch of the Bureau of Standards by the National Fire Proofing Co.

II. SCOPE OF THE TESTS

The tests made upon a material are, as a rule, those which are closely related to its intended use, and with this in mind the tests described in this paper are limited to those of compression and absorption. The total number of tests performed is approximately 250, of which the majority were of tiles in compression. Stress-strain readings were taken upon 114 of these for moduli of elasticity determinations. To establish the relationship existing between the strength and porosity about 70 absorption tests were made upon samples taken from tiles previously tested in compression.

The various sizes and types of tiles tested, arranged according to their structure, are shown in Figs. 1 and 2 (frontispiece) in which all the tiles are lying on edge. Also the tiles were graded according to their color as dark, medium, or light, corresponding to the variations produced by burning as described in the introduction. The tiles tested were made with few exceptions from clay of the buff-burning variety, and the colors given above indicate variations in the buff color; that is, they correspond respectively to dark buff, medium buff, and light buff.

III. GENERAL PROCEDURE OF TESTS

1. MEASURING AND WEIGHING OF TILES

It was necessary that an accurate determination be made of the net sectional area of each tile as a basis for the computation of the increments of loading and the unit loads. This was done by measuring the walls and partitions of the tiles with calipers and computing the sectional area from these measurements. The product of the sectional area taken laterally with respect to the cells and the length gave the actual volume of material in a tile. The actual volume of material in a tile divided into its weight gave the weight per cubic unit of the constituent material.

2. COMPRESSION TESTS

Previous to the actual testing, the tiles were capped with plaster of Paris to insure a uniform bearing in the testing machine. Plugs were also set in the tiles for compressometer readings. The type of compressometer used was the 8-inch Berry strain gage, and readings were taken with it near the four corners of every tile upon which stress-strain relations were desired. The testing machines used were of the Olsen universal type.

IV. DETAILS OF COMPRESSION TESTS

The sectional area and weight of each tile were determined as previously described. Then two small holes 8 inches from center to center were drilled with a small air-hammer drill near each corner of the tiles upon which stress-strain measurements were to be made. There were then placed in these holes, with plaster of Paris, small brass plugs three-sixteenths of an inch in diameter by three-eighths of an inch long, with appropriate holes for setting the gage.

The plaster of Paris caps were applied to each tile in the following manner: The surface of a smooth plate of steel was well coated with engine oil. Then a small quantity of a plastic mixture of plaster of Paris and water was spread on it in a space slightly larger than the end or face of the tile to be capped, and the tile was forced down into the mass. Care was taken that the axis of the tile remained vertical. The plaster mixture hardened in a short time, and the tile was slipped horizontally from the plate. The other end or opposite side was capped in the same manner. Special care was exercised to obtain caps of uniform thickness with smooth surfaces, and yet keep them as thin as possible.

In testing, each tile was placed in the machine on a hemispherical bearing block in such a manner that its vertical axis coincided with an imaginary vertical line passing through the center of the movable head. The tile and the upper part of the bearing block were rotated slowly as the movable head was brought in contact with the tile in order to secure the best bearing possible.

The speed of the moving head during a test was not less than 0.01 inch nor more than 0.05 inch per minute. The slowest speed was that of the 100 000-pound-capacity Olsen universal machine, and the fastest was that of the 600 000-pound-capacity Olsen universal machine.

No strain readings were taken in tests where the maximum height of the specimen was less than 10 inches, but where the specimen was 10 inches or more in height strain readings were taken with the 8-inch Berry gage, beginning with an initial load of 100 pounds per square inch of net sectional area and continued at increments of 500 pounds per square inch until failure was approached.

V. ABSORPTION TESTS

Three pieces were selected from each tile upon which the absorption determination was desired. Two of the three pieces were taken from opposite ends of the specimen and from different webs, and one from the middle portion. The pieces were approximately square and ranged from 12 to 20 square inches area of face and were free from cracks or fissures from the breaking in the compression test.

Preparatory to the absorption tests, all the specimens were first weighed and then dried over night in a gas oven at a temperature of about 120° C. After removal from the oven, the specimens were allowed to cool to room temperature and then were reweighed. As the second weighing closely checked the first in each case, they were immediately placed on a wire netting in a boiler, covered with distilled water, raised to the boiling point, and boiled for five hours. Then they were cooled in water to a final temperature of 10 to 15° C, removed from the water, and allowed to drain not more than one minute. The superficial water was removed with a towel, and the final weighing made at once.

The increase in weight of the piece after immersion times 100 divided by the dry weight gave the percentage of absorption

VI. RESULTS AND DISCUSSION OF TESTS

1. WEIGHT PER CUBIC FOOT

The actual weights of the individual tiles tested ranged according to their sizes from about 9 pounds for the 4 by 5 by 12 inch to approximately 50 pounds for the 12 by 12 by 12 inch type 3. Weights of these and intermediate types are shown in Table 2, column 4. The average weight per cubic foot of the constituent material of the tiles tested was determined to be approximately 124 pounds by using the net sectional area, the length, and the weight as the basis for the computations. The maximum variations from this average were 116 and 135 pounds, or 6.5 and 8.9 per cent, respectively.

These variations from the average are not the extremes to be expected by any means, but actually represent the range in this particular lot. The variations in weight depend upon differences in the porosity caused by a nonuniformity in mixing the clay, by differences in burning temperatures, and by changes in the amount of pressure to which the clay is subjected while being molded, for the greater the pressure the more dense or compact it will be, and vice versa. Also different clays in their natural state are quite variable in their densities within certain limits; consequently corresponding variations are to be expected in the weight of tiles made from them regardless of the other factors. However, the greatest variation in weight is produced by mixing sawdust in different percentages as desired with the clay before burning. This is especially true in the case of porous and semiporous tile, but does not relate to the results obtained upon the dense wall tiles of this series of tests. Thus, for other lots of tiles, the average might be expected to deviate from that given above in accordance with the kinds of clays used, the pressure applied during the molding, and the degree of artificial porosity.

2. COMPRESSION TESTS

Table 1 gives the general results of the compression tests of the various sizes and types of tiles, including the position in which tested, the color, the net sectional area, the load at incipient failure, the maximum load sustained, and the modulus of elasticity. The average maximum compressive strengths and the range in compressive strengths of every size and type of tile tested are shown diagrammatically in Figs. 3, 4, and 5. The average moduli and range in moduli in millions of pounds per square inch of the same tiles are shown diagrammatically in Figs. 7 and 8.

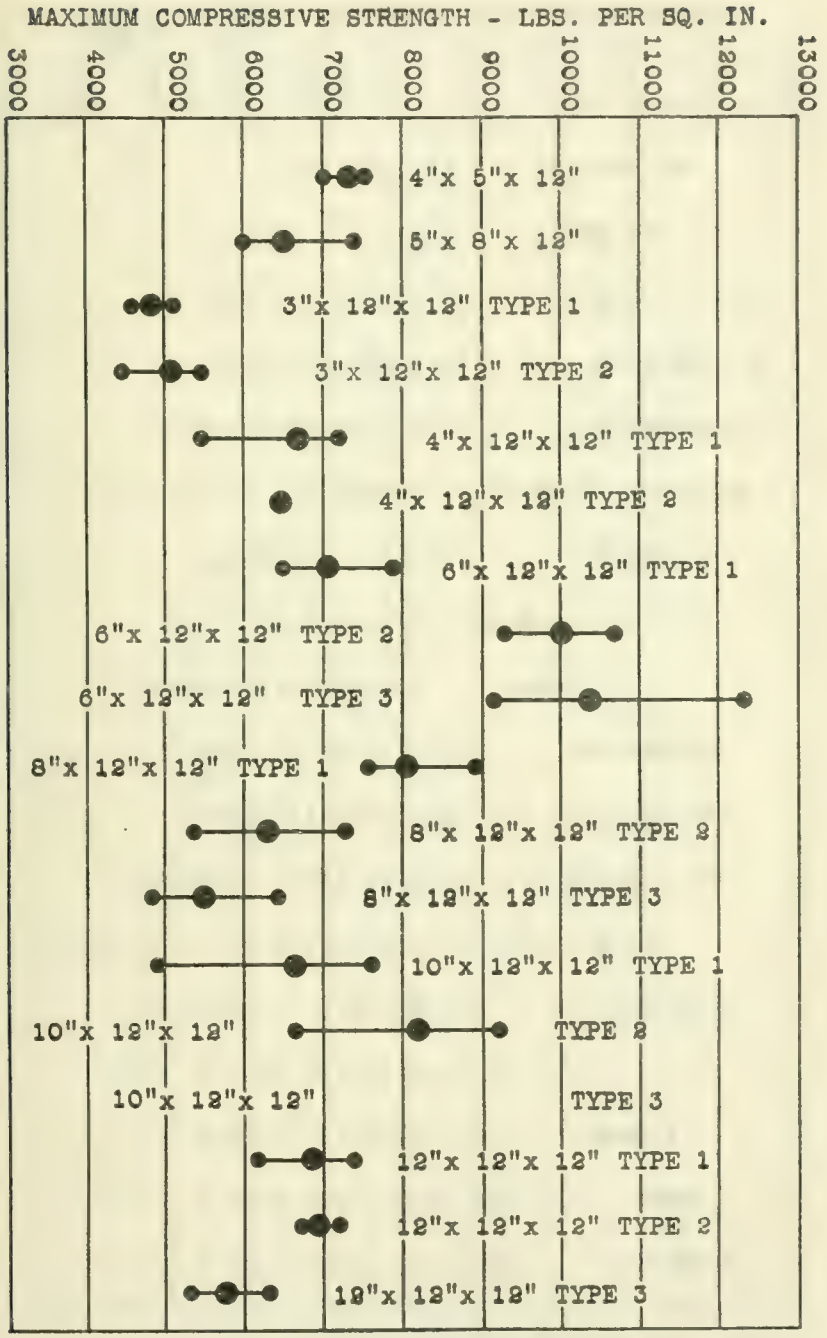


FIG. 3.—*Average and range of compressive strengths in pounds per square inch of net sectional area for tiles tested on end*

86063—18——2

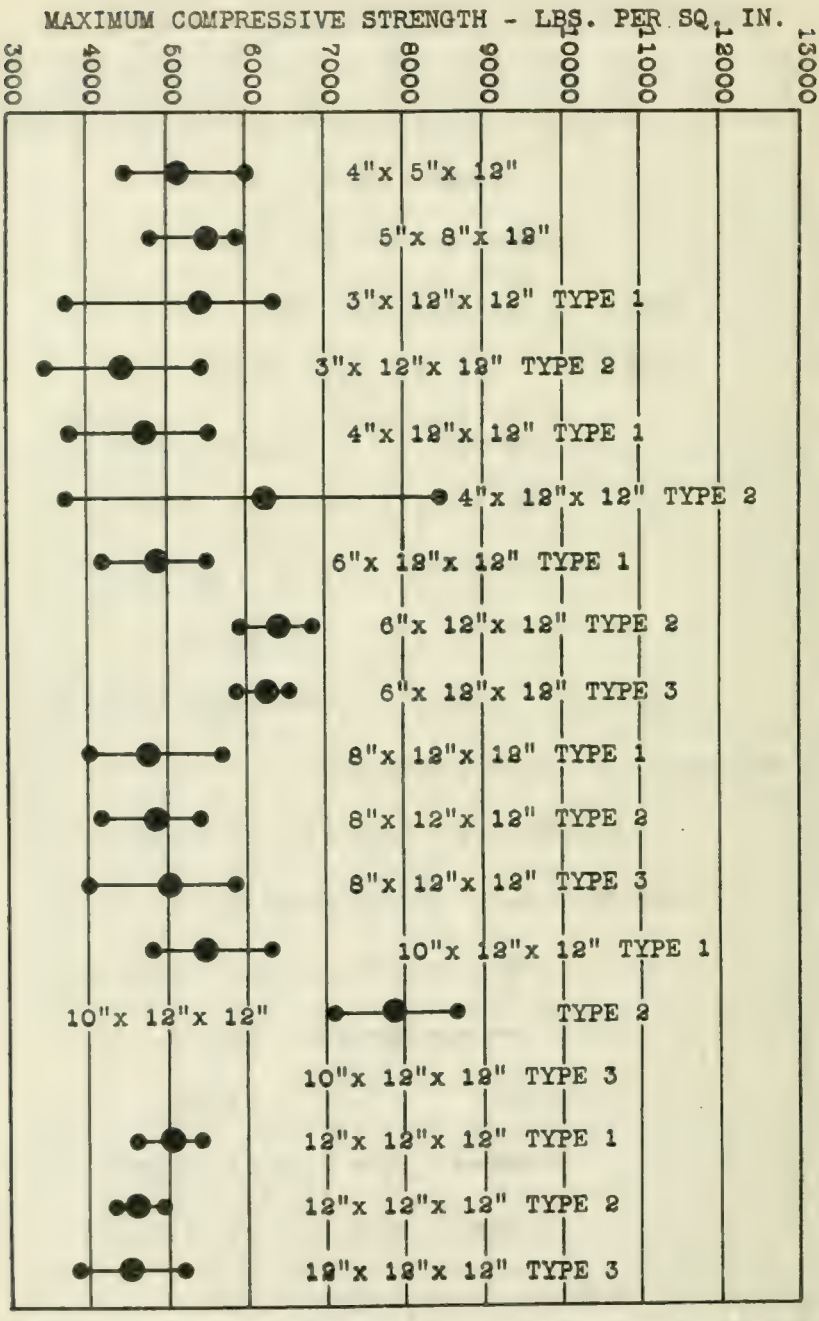


FIG. 4.—*Average and range of compressive strengths in pounds per square inch of net sectional area for tiles tested on edge*

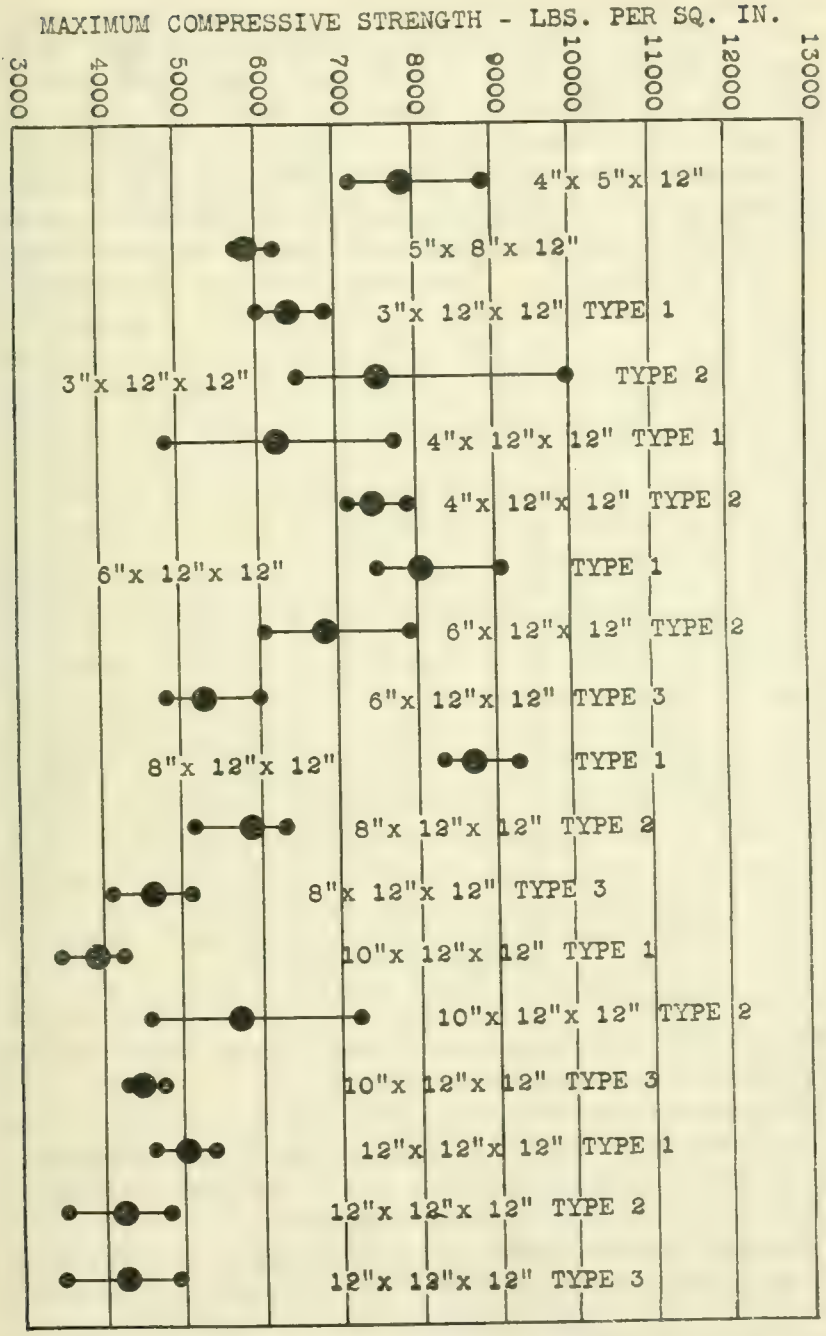


FIG. 5.—*Average and range of compressive strength in pounds per square inch for net sectional area of tiles tested flat*

Table 5 shows representative logs of compression tests upon tiles 10 by 12 by 12 inches, type 3, and 12 by 12 by 12 inches, type 1, respectively, and Fig. 6 shows the corresponding stress-strain curves. Columns 3, 4, 5, and 6 of Table 5 give the actual readings of the Berry strain gage at the four corners of the tiles, and columns 7, 8, 9, and 10 give the differences in the readings. The average of the differences, corrected for the constant of the gage, divided by the gage length in inches gave the strain which is recorded in column 13. These strains were used as abscissas in

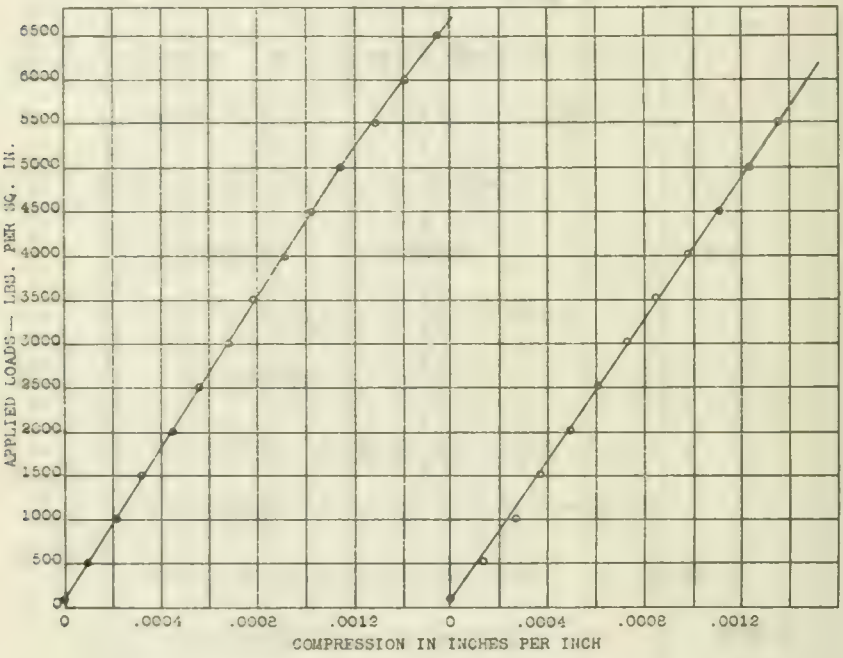


FIG. 6.—*Representative stress-strain curves of tiles.* (See Table 5)

plotting the stress-strain curves of which the ordinates were the applied loads in pounds per square inch.

The behavior of the other specimens was very similar to those shown; that is, the strain was approximately a linear function of the applied load from the initial load to the point of incipient failure always and often to the maximum load.

The term incipient failure is used to designate the point in the loading at which the first noticeable failure of any kind commences. Usually it was cracking accompanied by sudden and sharp reports. The exact load at incipient failure could not be definitely determined every time, but the loads given in the tables are well within 500 pounds per square inch of the truth.

The moduli of elasticity as given in Table 1 were obtained by calculation from the stress-strain data of the various tests. The modulus of elasticity is the ratio of the stress to the strain, or, in other words, the ratio of any ordinate of a stress-strain curve to the corresponding abscissa so long as the strain is a linear function of the stress. This condition held true in most of the tests to failure. The stress-strain curves of Fig. 6 are given as representative of the entire series, for those of the other tests were all similar to them.

By referring to these same curves of Fig. 6, it will be noticed that there is no well-defined proportional limit for tiles, when proportional limit is defined as the point where the ratio of the stress to the strain ceases to be a constant. This means that the increments of strain corresponding to equal increments of loading remain approximately the same throughout the entire range of the test. This indicates, in other words, that the flow of tile material under stress is governed by its constant modulus of elasticity almost to failure, which might be expected to occur without much warning in the way of a rapidly increasing strain such as is obtained in tests of steel and various other structural materials.

Table 4 gives a summary of the results of the compression tests, including the maximum negative percentage deviations of the loads at incipient failure, the maximum loads, and the modulus of elasticity from the respective average results for every size and type of tile tested. The maximum positive percentage deviations of the results are not given, for they are of little practical value, and at the same time they are from a builder's or designer's viewpoint on the side of safety. The maximum negative percentage deviations serve as a guide in the selection of the proper factor of safety, for it is essential from the standpoint of safety and economic design to know to what extent the properties of any one tile might possibly fall below the average of that particular type.

By referring to Table 1, columns 2 and 6, a comparison of the strengths of the tiles in the three positions may be made. From the size 4 by 5 by 12 inches to and including size 6 by 12 by 12 inches, type 1, the position of greatest strength was with the tiles flat, and from size 6 by 12 by 12 inches, type 2, to size 12 by 12 by 12 inches, type 3, it was with the tiles on end. But as the smaller size tiles are rarely used flat in practice, it may be said that they as well as the longer sizes develop a greater strength on end than in any other useful position. It is common practice to

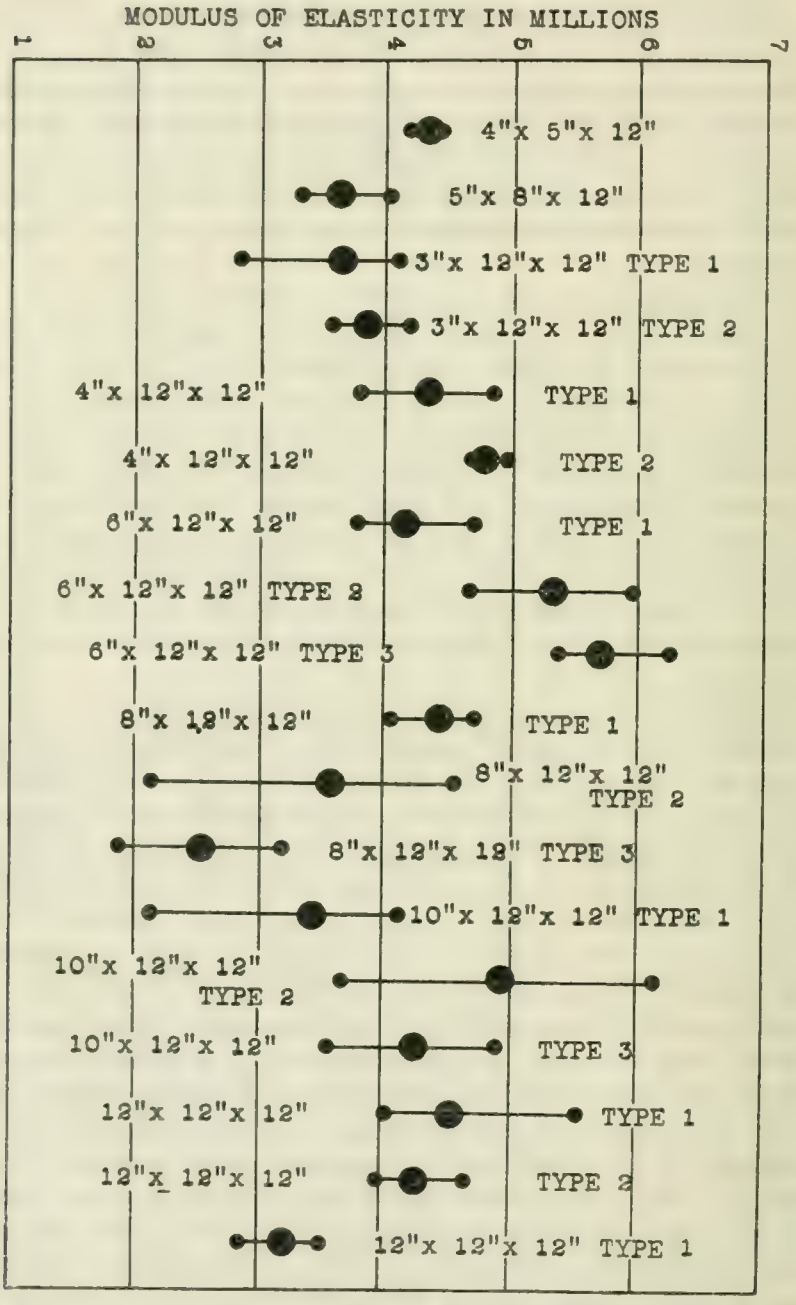


FIG. 7.—*Average and range of moduli of elasticity in units of million pounds per square inch of net sectional area for tiles tested on end*

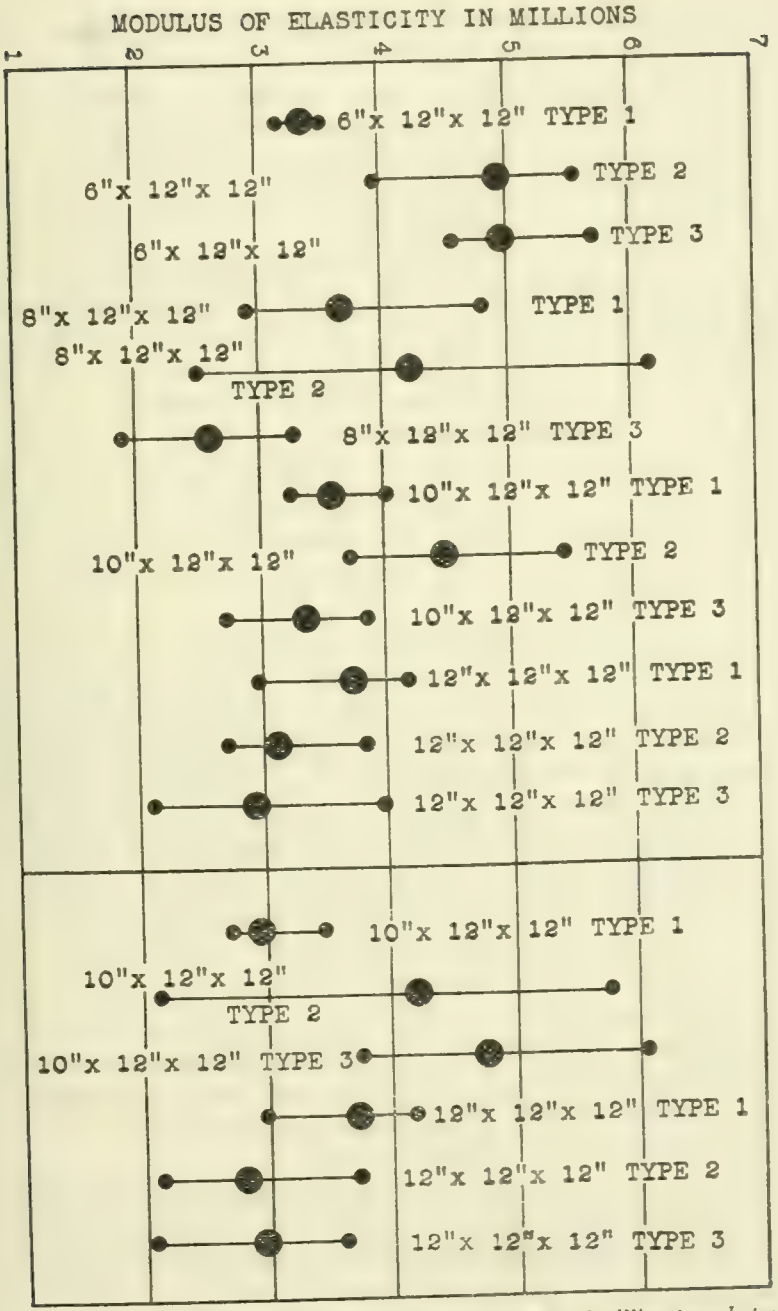


FIG. 8.—*Average and range of moduli of elasticity in units of million pounds per square inch of net sectional area for tiles tested on edge and flat*

lay tiles either on end or on edge in a building, but according to the results of this series of tests, excepting size 4 by 5 by 12 inches, it appears that an average of about 40 per cent more strength can be developed by laying the tiles on end rather than on edge; that is, by building all walls and other structures in such a manner that the load is applied to the tiles parallel to the flues or cells. The variations from this average are large and the strengths range from equality to a ratio of nearly three.

This increase in strength of the individual tiles by testing them on end was not due alone to a greater unit compressive strength in that position, but also to the fact that the load-bearing sectional area was greater in all the tiles tested with the exception of size 4 by 5 by 12 inches. The net sectional areas of all tiles tested are shown in column 4 of Table 1. Only in the case of the 12 by 12 by 12 inch tiles would it ever be feasible to consider laying tiles flat in a structure because of the enormous waste of material for smaller sizes, but, if the 12 by 12 by 12 inch tiles were laid flat, it would be uneconomical of strength, for twice the strength could be developed by laying them on end.

There was found to be no definite relation between the loads at the incipient failure and the maximum loads sustained by the tiles; however, it may be said that the fewer the partitions of a tile the more nearly the load at incipient failure approaches the maximum load, and vice versa. This is usually true because actual failure must be imminent in the case of tiles of few partitions when incipient failure occurs. And when failure begins, the maximum load is quickly reached, for the failure of one wall or partition decreases the resistance of the tile to a great extent and causes a proportional increase in the stress upon the other walls, which in turn increases the liability of their immediate failure. When a tile has a large number of partitions and its external walls are divided into numerous sections by intercepting partitions, the failure of one or two of them would not greatly influence the remaining sound sections of walls and partitions, and would not necessarily expedite the ultimate failure.

The actual tests indicate that, on the average, incipient failure occurs at approximately 76 per cent of the maximum load, but wide variations from this average were obtained. For instance, in one case the incipient failure occurred at only 19 per cent of the maximum load, and in several tests no incipient failure was perceived before the maximum load was reached.

By reference to the curves of Fig. 9, it will be noticed that there is a general relation between the modulus of elasticity and the maximum compressive strength of the tiles. Although there are wide variations, it appears that the higher the modulus the higher the maximum compressive strength will be also. This is true in about the same proportion for any position in which the tiles are tested, for it will be noticed that the curves of Fig. 9 are approximately parallel.

The relationship existing between the compressive strength of the tiles and their colors is not very definite, but the following table shows a summary of the results obtained in this series of tests:

Compressive Strength in Pounds per Square Inch

Color	Average	Range
Dark buff	7510	4650 to 10 700
Medium buff	7400	4500 to 12 360
Light buff	5530	4820 to 7 500

This comparison is based upon results of only the tiles tested on end, because if the result of tests on edge and on flat were used they would introduce complicating factors, especially the variable heights of the test specimens. The results indicate that the dark and the medium-burned tiles are of nearly the same strength while the light-burned tiles are on the average 25 or 30 per cent lower.

The relation of the colors of the tiles to their moduli of elasticity is in about the same proportion as between their colors and compressive strengths. The following is a summary of the results:

Modulus of Elasticity

Color	Average	Range
Dark buff	4 519 000	2 830 000-6 160 000
Medium buff	4 352 000	3 190 000-6 260 000
Light buff	3 074 000	1 880 000-4 170 000

These results are also based only upon tests of tile on end. The light-burned tiles, it may be noticed, develop moduli of only about 60 per cent of those of the dark and medium burned.

3. ABSORPTION TESTS

Absorption tests were not made upon all the tile upon which compression tests were made, but some were made upon every size and type of the same lot. Those selected were tested in

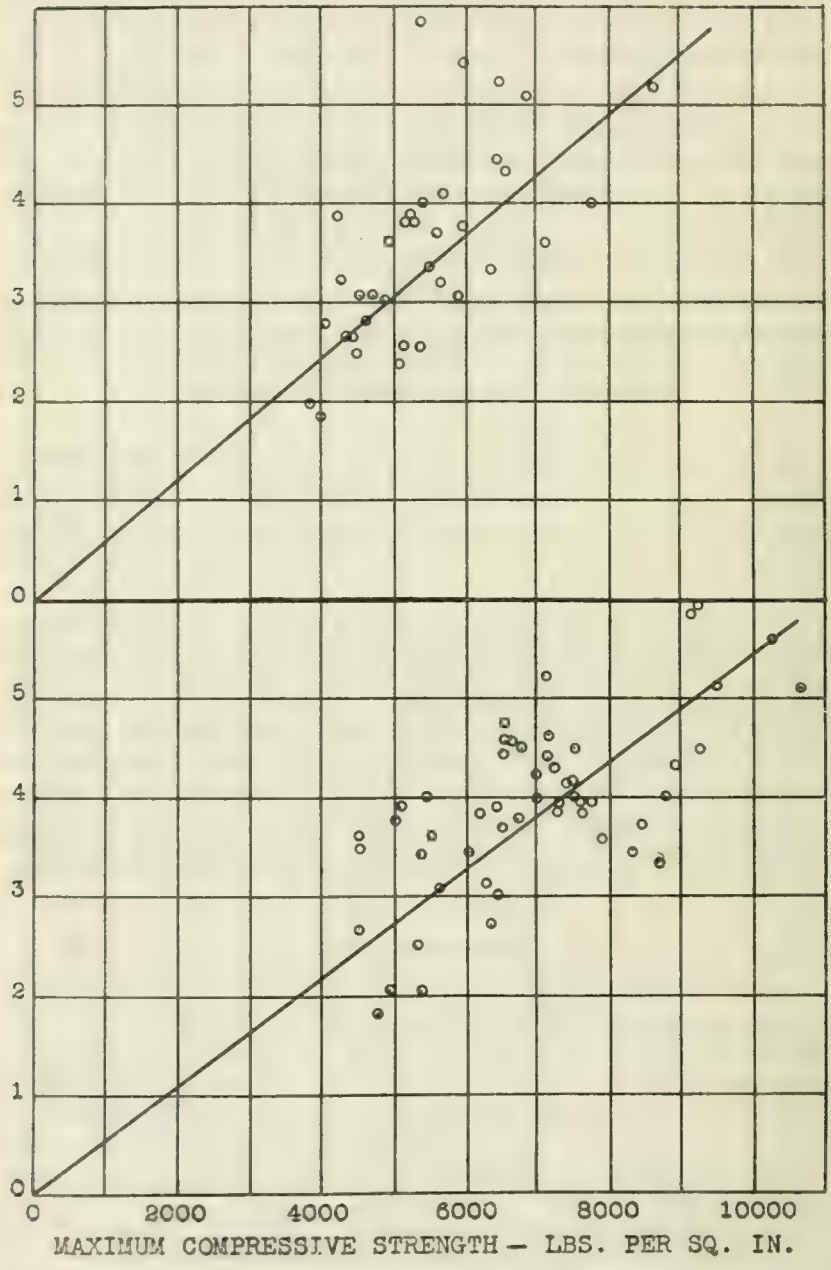


FIG. 9.—*Relation of moduli of elasticity in units of million pounds per square inch of net sectional area for tiles tested on edge (upper curve) and on end (lower curve) to the maximum compressive strengths*

compression without taking strain readings, and samples were chosen from them and tested as previously described in the "General procedure of tests." The results of these tests are given in Table 2. Table 3 shows results of similar absorption tests, but the samples were not from the same lot of tiles as those of Table 2 and strain readings were taken during the compression tests. Fig. 10 shows the relation curve of percentage absorptions to the maximum compressive strengths of the tiles. This curve was plotted according to the results given in Tables 2 and 3, and shows the above relation only in a general way, for the results have such large percentage variations that a definite law can not be drawn. However, it may be said that there is a tendency for the maximum compressive strength to vary inversely with the percentage of absorption. This is because the absorption is a function of the porosity as is also the maximum compressive strength.

The following summary of results shows the relationship of the percentages of absorption to the colors of the tiles:

Per Cent Absorption

Color	Average	Range
Dark buff	5.3	3.8– 6.7
Medium buff	7.3	5.1– 8.7
Light buff	9.3	7.2–10.9

Although there is overlapping in the percentages of absorption from one color to another, it may be said that, in general, judging by these results, the darker the tiles the lower the percentages of absorption are likely to be. However, as previously stated, this is not always true, because either the material or the artificial color of the tiles may often be deceptive in this respect, and it should be remembered that the tiles tested in this series were made of buff-burning clay.

VII. GENERAL SUMMARY OF TESTS

The following conclusions, drawn from the tests collated in this paper, are based directly upon the results obtained, and their applicability to other tiles must, of necessity, be dependent upon similar conditions. In drawing independent conclusions and making applications it should be borne in mind that conditions in practice are quite different from those of the laboratory, that there are many grades and varieties of tiles, and that wide variations in

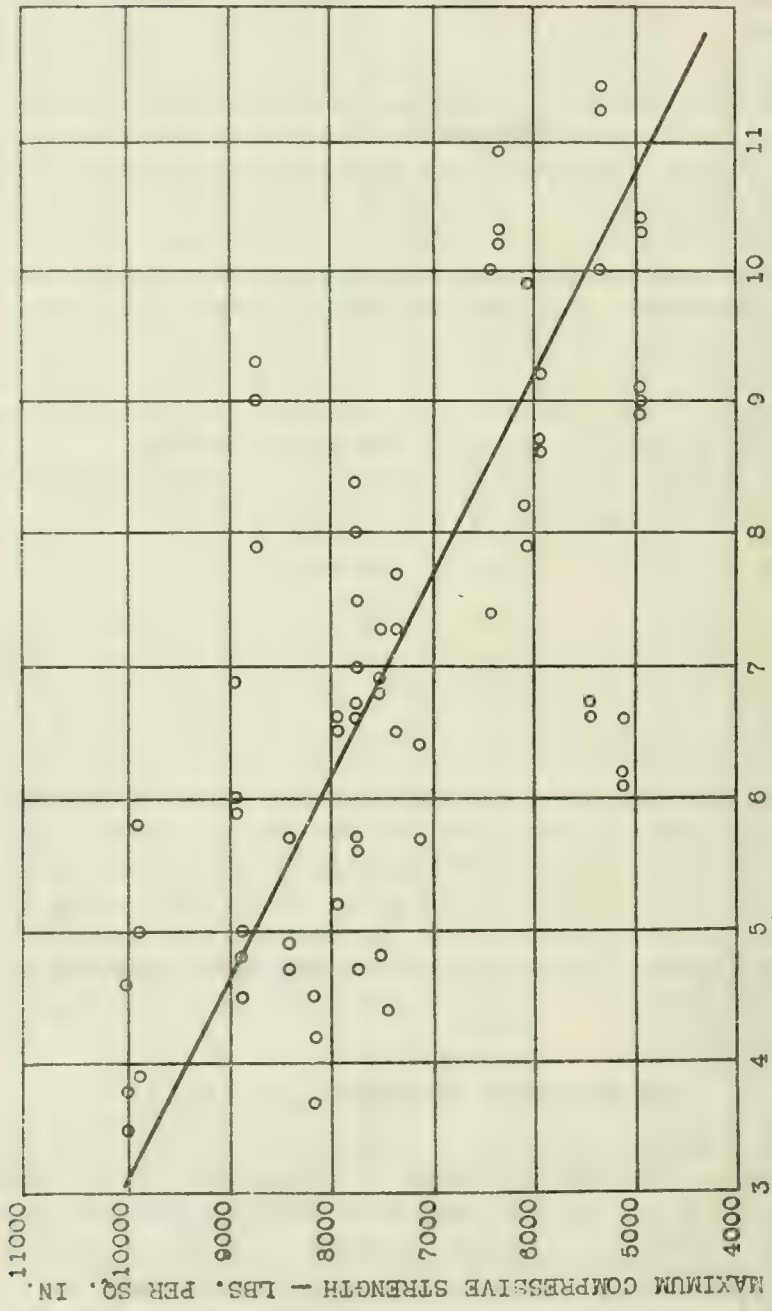


FIG. 10.—*Relation of percentages of absorption of tiles to their maximum compressive strengths.* (See Tables 2 and 3)

the strength properties are possessed by tiles which are apparently similar.

1. The weight of the tiles per cubic unit varies greatly with the porosity of the constituent material.

2. The porosity of the constituent material of tiles is dependent upon the clays used, the percentage of sawdust mixed with the clay before burning, the pressure to which it is subjected during molding, and the degree of burning.

3. The color of the same tile material is subject to change during the burning process, and the resulting tiles may vary in color from very light to very dark shades, depending upon the temperature reached and the time of burning.

4. The strain produced by loading a tile is approximately a linear function of the applied load until failure is reached, or, in other words, the modulus of elasticity of a tile is nearly constant until failure.

5. There is no definite elastic limit for tile; that is, the proportional limit is usually coincident with failure.

6. An incipient failure is not always indicative that the ultimate failure is being approached, as it often occurs early in a test.

7. In general a tile develops greatest compressive strength when laid on end.

8. The tile having the highest modulus of elasticity may not have the greatest strength, but, in general, if the modulus is high, it is to be expected that the compressive strength will also be relatively high.

9. The dark and medium buff tiles have approximately the same relative compressive strengths and moduli of elasticity. The average compressive strength of tiles of these colors tested on end is about 7500 pounds per square inch, with a possible range from 4500 to 12 000 pounds per square inch, and the average modulus of elasticity is about 4 430 000 pounds per square inch, with a possible range from 2 800 000 to 6 200 000 pounds per square inch. The same properties of light-burned tiles are on an average about 30 per cent lower.

10. The maximum compressive strength of tiles varies approximately inversely with the percentage of absorption.

11. The percentages of absorption of tiles vary with the color. In general, the darker the tiles the lower the percentage of absorption is likely to be.

TABLE 1.—Compression Tests of Hollow Building Tile

Size of tile	How tested	Color	Net section area, in square inches	Load at incipient failure, in pounds per square inch	Maximum load		Modulus of elasticity, in pounds per square inch
					Pounds	Pounds per square inch	
Type 1:							
4 by 5 by 12 inches	End	Light	10.11	5500	75 825	7500	4 170 000
	Do	Dark	9.98	6500	74 850	7500	4 370 000
	Do	do	9.77	6000	68 390	7000	4 410 000
Average				6000	73 020	7330	4 317 000
	Edge	Medium	15.37	2000	69 000	4490	.....
	Do	do	15.37	.....	76 700	4990	.....
	Do	do	15.25	2500	91 500	6000	.....
Average				2250	79 070	5160	.....
	Flat	Medium	14.16	7000	102 350	7230	.....
	Do	do	14.00	7000	104 800	7490	.....
	Do	do	14.04	.....	125 640	8950	.....
Average				7000	110 930	7890	.....
5 by 8 by 12 inches	End	Medium	16.79	4500	123 420	7350	4 020 000
	Do	do	16.79	5500	100 740	6000	3 580 000
	Do	do	16.79	5000	104 380	6220	3 300 000
Average				5000	109 510	6520	3 633 000
	Edge	Medium	13.55	2500	65 400	4826	.....
	Do	Dark	14.52	2400	85 850	5910	.....
	Do	Medium	13.55	1640	79 800	5890	.....
Average				2180	77 020	5540	.....
	Flat	Medium	23.08	.....	135 000	5850	.....
	Do	do	22.25	.....	128 000	5780	.....
	Do	Dark	23.30	.....	143 350	6150	.....
Average				.....	135 450	5930	.....
3 by 12 by 12 inches	End	Light	16.02	3000	80 580	5030	4 090 000
	Do	Dark	16.24	2500	74 000	4560	2 830 000
	Do	Light	14.99	3500	74 950	5000	4 000 000
Average				3000	76 510	4860	3 640 000
	Edge	Medium	10.32	3000	65 500	6350	.....
	Do	do	10 32	2420	64 050	6210	.....
	Do	do	11.16	2150	41 500	3720	.....
Average				2520	57 020	5430	.....
	Flat	Medium	21.53	.....	137 400	[illegible]	.....
	Do	do	21.99	.....	152 100	6920	.....
	Do	do	21.09	.....	128 300	6080	.....
Average				.....	139 270	6460	.....
Type 2: 3 by 12 by 12 inches	End	Dark	17.42	3500	95 370	5470	4 180 000
	Do	Medium	17.23	3500	77 535	4500	3 760 000
	Do	Dark	16.89	3500	89 680	5310	3 570 000
Average				3500	87 530	5100	3 837 000
	Edge	Light	10.71	3540	58 000	5420	.....
	Do	Medium	11.13	3140	49 450	4440	.....
	Do	Dark	10.85	3480	37 800	3480	.....
Average				3390	48 420	4450	.....
	Flat	Light	21.60	.....	141 550	6550	.....
	Do	Medium	21.72	.....	154 300	7100	.....
	Do	Dark	21.03	.....	210 000	9990	.....
Average				.....	168 620	7550	.....

TABLE 1.—Compression Tests of Hollow Building Tile—Continued

Size of tile	How tested	Color	Net section area, in square inches	Load at incipient failure, in pounds per square inch	Maximum load		Modulus of elasticity, in pounds per square inch
					Pounds	Pounds per square inch	
Type 1: 4 by 12 by 12 inches	End	Dark	19.44	.....	106 920	5500	3 780 000
	Do	do	18.79	3500	135 260	7200	4 840 000
	Do	do	20.70	3000	154 700	7470	4 360 000
Average				3250	132 290	6720	4 327 000
	Edge	Light	12.47	1120	47 000	3770	.....
	Do	Medium	12.24	1230	67 530	5520	.....
	Do	Dark	12.47	1450	61 650	4940	.....
Average				1270	58 730	4740	.....
	Flat	Medium	21.84	.....	170 130	7790	.....
	Do	Dark	23.06	.....	112 030	4860	.....
	Do	Light	23.26	.....	146 020	6280	.....
Average				.....	142 730	6310	.....
Type 2: 4 by 12 by 12 inches	End	Medium	20.63	5000	134 095	6500	4 640 000
	Do	do	20.58	3000	133 770	6500	4 970 000
	Do	Dark	20.27	4000	132 000	6510	4 750 000
Average				4000	133 280	6500	4 787 000
	Edge	Light	13.26	.....	49 000	3700	.....
	Do	Medium	12.55	5500	105 880	8440	.....
	Do	Dark	12.72	3300	85 650	6730	.....
Average				4400	80 180	6290	.....
	Flat	Medium	25.74	.....	185 070	7190	.....
	Do	do	25.58	.....	202 000	7900	.....
	Do	Dark	25.80	.....	192 120	7450	.....
Average				.....	193 060	7510	.....
Type 1: 6 by 12 by 12 inches	End	Dark	22.80	6000	181 080	7940	3 760 000
	Do	do	24.90	5000	170 000	6830	4 720 000
	Do	Medium	25.56	6000	166 140	6500	3 910 000
Average				5670	172 410	7090	4 130 000
	Edge	Medium	13.56	4000	66 770	4920	3 180 000
	Do	do	12.12	.....	66 680	5500	3 510 000
	Do	do	13.06	3830	55 040	4210	3 430 000
Average				3920	62 830	4880	3 373 000
	Flat	Medium	23.86	.....	181 340	7600	.....
	Do	do	23.96	.....	218 650	9130	.....
	Do	do	24.62	.....	185 300	7530	.....
Average				.....	195 100	8090	.....
Type 2: 6 by 12 by 12 inches	End	Dark	29.63	6500	305 180	10 300	5 960 000
	Do	do	31.32	8800	291 340	9300	4 700 000
	Do	do	30.03	9500	321 860	10 700	5 370 000
Average				8270	306 130	10 100	5 343 000
	Edge	Light	19.34	3500	115 530	5970	3 970 000
	Do	Dark	18.75	3500	120 300	6420	5 540 000
	Do	do	19.06	3000	130 860	6860	5 330 000
Average				3330	122 230	6420	4 947 000
	Flat	Medium	25.38	3300	201 820	7950	.....
	Do	do	26.11	2300	170 080	6510	.....
	Do	do	25.85	2320	158 660	6130	.....
Average				2640	176 850	6860	.....

TABLE 1.—Compression Tests of Hollow Building Tile—Continued

Size of tile	How tested	Color	Net section area, in square inches	Load at incipient failure, in pounds per square inch	Maximum load		Modulus of elasticity, in pounds per square inch
					Pounds	Pounds per square inch	
Type 3: 6 by 12 by 12 inches	End	Medium	33.78	5500	310 300	9190	6 260 000
	Do	do	33.46	6000	413 540	12 360	5 430 000
	Do	Dark	33.55	6500	319 100	9510	5 380 000
Average				6000	347 650	10 350	5 690 000
	Edge	Dark	22.13	3000	142 900	6460	4 700 000
	Do	Medium	22.32	3500	145 860	6540	4 550 000
	Do	Dark	21.69	1500	128 780	5930	5 700 000
Average				2670	139 150	6310	4 983 000
	Flat	Medium	29.87	.....	180 320	6040	.....
	Do	Dark	29.09	1030	151 180	5200	.....
	Do	do	30.45	920	147 500	4840	.....
Average				975	156 330	5360	.....
Type 1: 8 by 12 by 12 inches	End	Dark	26.92	.....	241 400	8970	4 540 000
	Do	Medium	27.46	3500	211 920	7720	4 070 000
	Do	do	27.61	6500	208 200	7540	4 700 000
Average				5000	220 510	8080	4 437 000
	Edge	Medium	12.19	3940	56 180	4610	3 240 000
	Do	Dark	11.26	4170	64 300	5710	4 810 000
	Do	Medium	12.00	2000	48 410	4030	2 930 000
Average				3370	56 300	4780	3 660 000
	Flat	Medium	23.66	.....	201 340	8510	.....
	Do	Dark	22.93	.....	192 200	8382	.....
	Do	do	23.34	.....	217 500	9320	.....
Average				.....	203 680	8740	.....
Type 2: 8 by 12 by 12 inches	End	Dark	37.49	2000	272 920	7280	4 540 000
	Do	Medium	39.54	4000	251 340	6360	4 070 000
	Do	Light	44.52	.....	240 240	5400	2 140 000
Average				3000	254 830	6350	3 583 000
	Edge	Dark	20.73	2000	112 540	5430	6 160 000
	Do	Medium	20.27	1500	85 250	4210	4 070 000
	Do	Light	19.74	.....	99 360	5030	2 460 000
Average				1750	99 050	4890	4 230 000
	Flat	Dark	27.76	.....	177 000	6378	.....
	Do	Medium	27.56	3050	173 800	6310	.....
	Do	do	28.41	.....	147 640	5200	.....
Average				3050	166 150	5960	.....
Type 3: 8 by 12 by 12 inches	End	Light	40.22	3250	211 390	5260	2 660 000
	Do	Medium	42.08	.....	270 860	6440	3 190 000
	Do	Light	41.82	.....	201 700	4820	1 880 000
Average				3250	227 980	5510	2 573 000
	Edge	Light	20.99	.....	123 380	5880	3 240 000
	Do	do	21.59	.....	86 360	4000	1 910 000
	Do	do	21.16	.....	108 790	5140	2 660 000
Average				.....	106 180	5010	2 603 000
	Flat	Medium	31.03	2000	143 850	4635	.....
	Do	do	30.20	1860	124 940	4137	.....
	Do	Light	30.39	3900	157 520	5183	.....
Average				2590	142 100	4650	.....

TABLE 1. Compression Tests of Hollow Building Tile—Continued

Size of tile	How tested	Color	Net section area, in square inches	Load at incipient failure, in pounds per square inch	Maximum load		Modulus of elasticity, in pounds per square inch
					Pounds	Pounds per square inch	
Type 1: 10 by 12 by 12 inches.	End	Light	39.00		193 600	4960	2 190 000
	Do	Dark	37.36		281 630	7540	4 130 000
	Do	Medium	37.82	3500	287 800	7610	3 970 000
Average				3500	254 340	6700	3 430 000
	Edge	Medium	20.76		108 100	5210	4 020 000
	Do	do	20.47	3950	98 340	4800	3 240 000
	Do	do	20.57	3080	129 780	6310	3 480 000
Average				3480	112 410	5440	3 580 000
	Flat	Medium	21.55		92 310	4280	3 450 000
	Do	do	21.53	2500	88 860	4130	2 720 000
	Do	do	21.20		74 450	3510	2 720 000
Average				2500	85 210	3970	2 963 000
Type 2: 10 by 12 by 12 inches.	End	Medium	41.98	3500	279 140	6650	4 810 000
	Do	do	43.00	4500	379 500	8830	3 660 000
	Do	Dark	42.76	3500	389 840	9120	6 160 000
Average				3830	349 490	8200	4 877 000
	Edge	Dark	20.69	3000	178 820	8640	5 430 000
	Do	do	20.57	1500	159 480	7750	4 180 000
	Do	Medium	20.79	5000	148 700	7150	3 710 000
Average				3170	162 330	7850	4 440 000
	Flat	Light	28.48	4500	131 450	4620	2 140 000
	Do	Dark	25.69	7200	186 400	7260	4 700 000
	Do	do	26.00	2500	141 200	5430	5 750 000
Average				4730	153 020	5770	4 237 000
Type 3: 10 by 12 by 12 inches.	End	Medium	46.38	4500	407 860	8792	4 230 000
	Do	do	48.60	6500	407 440	8382	3 930 000
	Do	do	48.17		419 040	8706	3 550 000
Average				5500	411 450	8630	3 903 000
	Edge	Medium	22.87		128 440	5614	3 400 000
	Do	Dark	22.81	3500	127 920	5607	3 866 000
	Do	Medium	23.58		127 240	5394	2 720 000
Average				3500	127 870	5540	3 327 000
	Flat	Medium	30.96	2000	133 900	4330	3 760 000
	Do	do	30.19	2000	144 420	4780	4 490 000
	Do	Dark	30.23	1500	131 060	4340	6 060 000
Average				1830	136 460	4480	4 770 000
Type 1: 12 by 12 by 12 inches.	End	Dark	45.10	5000	277 520	6153	4 030 000
	Do	Medium	47.38	6000	348 000	7350	4 070 000
	Do	do	42.52	5200	303 200	7130	5 540 000
Average				5400	309 570	6880	4 547 000
	Edge	Medium	22.26	2500	115 800	5202	3 970 000
	Do	do	22.68	3500	122 580	5402	4 180 000
	Do	do	22.59	1500	105 020	4649	2 980 000
Average				2500	114 470	5080	3 710 000
	Flat[a]						

[a] Same as on edge.

TABLE 1. Compression Tests of Hollow Building Tile Continued

Size of tile	How tested	Color	Net section area, in square inches	Load at incipient failure, in pounds per square inch	Maximum load		Modulus of elasticity, in pounds per square inch.
					Pounds	Pounds per square inch	
Type 2: 12 by 12 by 12 inches.	End	Medium	47.91	5500	334 440	6980	4 180 000
	Do	do	47.25	3500	318 450	6740	3 970 000
	Do	do	45.78	4500	329 960	7206	4 650 000
	Average			4500	327 620	6980	4 267 000
	Edge	Medium	21.69	2500	106 950	4932	3 820 000
	Do	do	22.09	1500	96 660	4376	2 820 000
	Do	do	22.03		100 000	4540	2 690 000
	Average			2000	101 200	4620	3 110 000
	Flat	Medium	28.44		126 300	4442	2 590 000
	Do	do	27.96	2500	99 690	3565	2 140 000
	Do	do	27.64		133 980	4848	3 710 000
	Average			2500	119 990	4290	2 813 000
Type 3: 12 by 12 by 12 inches.	End	Light	55.69	5500	353 220	6342	2 870 000
	Do	do	55.30	2000	312 500	5650	3 240 000
	Do	do	55.64	3500	296 200	5324	3 500 000
	Average			3670	320 640	5770	3 203 000
	Edge	Light	30.91	3400	119 620	3870	2 090 000
	Do	do	30.27	2400	135 700	4483	2 770 000
	Do	Medium	28.18	2000	146 190	5188	3 970 000
	Average			2600	133 840	4510	2 943 000
	Flat	Medium	28.30	4000	126 550	4472	3 600 000
	Do	Light	29.97	1670	104 315	3481	2 090 000
	Do	Medium	29.37	2200	146 500	4988	3 240 000
	Average			2620	125 790	4310	2 977 000

TABLE 2.—Absorption Tests

Size of original tile	Color	Net section area, in square inches	Weight, in pounds	Load at incipient failure, in pounds per square inch	Maximum load		Absorption per cent
					Pounds	Pounds per square inch	
Type 2:							
4 by 5 by 12 inches	Dark	10.9	9.25	[illegible]	58 950	5420	[illegible]
	Medium	10.6	9.12	5750	68 000	6420	8.6
Average		10.8	9.19	4940	[illegible]	5920	7.7
4 by 12 by 12 inches	Dark	20.8	19.40	[illegible]	[illegible]	7980	6.1
	Do	22.8	19.60	[illegible]	177 300	[illegible]	6.0
	Medium	22.3	19.38	5160	[illegible]	7100	6.2
Average		22.0	19.46	8990	167 250	7620	6.1
Type 3:							
6 by 12 by 12 inches	Dark	37.6	32.70	7030	376 000	10 000	3.8
	Medium	38.8	31.65	6700	284 000	8400	5.1
	Dark	37.6	31.70	7580	335 000	8920	3.8
Average		38.0	32.02	7100	331 [illegible]	9110	4.3
8 by 12 by 12 inches	Light	44.7	36.80	6260	[illegible]	6350	10.4
	Medium	42.7	37.20	8630	[illegible]	8770	8.7
	Light	42.7	35.65	5330	[illegible]	5830	10.9
Average		43.4	36.55	[illegible]	295 [illegible]	6820	10.0
Type 1: 12 by 12 by 12 inches	Medium	44.2	39.50	5750	[illegible]	8980	6.3
	Dark	46.6	40.15	4200	[illegible]	7780	6.3
	Medium	45.5	39.90	[illegible]	352 500	7750	7.8
Average		45.4	39.85	5340	370 500	8170	6.8
Type 3: 12 by 12 by 12 inches	Medium	56.6	49.30	4240	341 800	6040	8.7

TABLE 3.—Absorption Tests

Size of original tile	Color	Net section area, in square inches	Load at incipient failure, in pounds per square inch	Maximum load		Modulus of elasticity, in pounds per square inch	Absorption per cent
				Pounds	Pounds per square inch		
Type 3:							
6 by 12 by 12 inches	Dark	33.8	7000	334 900	9910	5 130 000	4.9
	Do	34.1	5000	279 300	8180	4 580 000	4.6
	Medium	33.7	4000	252 300	7480	2 700 000	4.4
Average		33.9	5330	288 830	8520	4 136 700	4.6
8 by 12 by 12 inches	Medium	42.7	3000	217 800	5100	3 450 000	6.3
	Do	43.9	4940	216 550	4940	3 180 000	9.0
	Do	46.5	4980	232 000	4980	2 700 000	9.9
Average		43.7	4310	222 120	5010	3 110 000	8.4
12 by 12 by 12 inches	Light	58.1	5000	426 200	7340	3 400 000	7.2
	Do	63.8	5000	379 200	5940	3 570 000	8.8
	Medium	56.9	5500	427 440	7520	3 850 000	6.3
Average		59.6	5170	410 950	6930	3 606 700	7.4

TABLE 4.—Summary of Results of Compression Tests

Size of tile	How tested	Average load at incipient failure, in pounds per square inch	Maximum negative deviation from the average per cent	Average maximum load, in pounds per square inch	Maximum negative deviation from the average per cent	Average modulus of elasticity, in pounds per square inch	Maximum negative deviation from the average per cent
Type 1:							
4 by 5 by 12 inches	End	5580	26.0	6770	19.9	4 317 000	3.4
	Edge	2250	11.1	5160	13.0	.....	.....
	Flat	7000	0.0	7890	8.4	.....	.....
5 by 12 by 12 inches	End	5000	10.0	6520	8.0	3 633 000	9.2
	Edge	2180	24.8	5540	12.8	.....	.....
	Flat	.....	.....	5930	1.3	.....	.....
3 by 12 by 12 inches	End	3000	16.7	4860	6.2	3 640 000	22.2
	Edge	2520	14.7	5430	31.5	.....	.....
	Flat	.....	.....	6460	5.9	.....	.....
Type 2: 3 by 12 by 12 inches	End	3500	0.0	5100	11.8	3 837 000	7.0
	Edge	3390	6.3	4450	21.8	.....	.....
	Flat	.....	.....	7550	13.3	.....	.....
Type 1: 4 by 12 by 12 inches	End	3250	8.3	6720	18.2	4 327 000	12.6
	Edge	1270	11.8	4740	20.4	.....	.....
	Flat	.....	.....	6310	23.0	.....	.....
Type 2: 4 by 12 by 12 inches	End	6495	53.8	7060	7.9	4 787 000	3.1
	Edge	4400	25.0	6290	4.1	.....	.....
	Flat	.....	.....	7510	4.3	.....	.....
Type 1: 6 by 12 by 12 inches	End	5670	13.4	7090	8.3	4 130 000	9.0
	Edge	3920	2.3	4880	13.7	3 373 000	5.1
	Flat	.....	.....	8090	6.9	.....	.....
Type 2: 6 by 12 by 12 inches	End	8270	21.4	10 100	7.9	5 343 000	12.0
	Edge	3330	10.0	6420	7.0	4 947 000	19.8
	Flat	2640	12.9	6860	10.6	.....	.....
Type 3: 6 by 12 by 12 inches	End	6140	34.8	9330	19.8	4 913 000	45.0
	Edge	2670	43.8	6310	6.0	4 983 000	8.7
	Flat	975	5.6	5360	9.7	.....	.....
Type 1: 8 by 12 by 12 inches	End	5000	30.0	8080	6.4	4 437 000	8.3
	Edge	3370	40.7	4780	15.7	3 660 000	19.9
	Flat	.....	.....	8740	4.1	.....	.....
Type 2: 8 by 12 by 12 inches	End	3000	33.3	6350	15.0	3 583 000	40.3
	Edge	1750	14.3	4890	13.6	4 230 000	41.8
	Flat	3050	.....	5960	12.7	.....	.....
Type 3: 8 by 12 by 12 inches	End	4770	37.2	5750	16.2	2 841 500	33.8
	Edge	.....	.....	5010	20.2	2 603 000	26.6
	Flat	2590	28.2	4650	11.1	.....	.....
Type 1: 10 by 12 by 12 inches	End	3500	.....	6700	26.0	3 430 000	36.2
	Edge	3480	13.8	5440	11.8	3 580 000	8.9
	Flat	2500	.....	3970	11.6	2 963 000	8.2
Type 2: 10 by 12 by 12 inches	End	3830	8.6	8200	18.9	4 877 000	25.0
	Edge	3170	52.6	7850	8.9	4 440 000	16.4
	Flat	4730	4.7	5770	20.0	4 237 000	49.5
Type 3: 10 by 12 by 12 inches	End	5500	18.2	8630	28.8	3 903 000	9.0
	Edge	.....	.....	5540	2.6	3 327 000	18.2
	Flat	1830	18.0	4480	3.3	4 770 000	21.2
Type 1: 12 by 12 by 12 inches	End	5370	21.8	7530	18.3	4 547 000	11.4
	Edge	2500	40.0	5080	8.5	3 710 000	19.7
	Flat	2500	40.0	5080	8.5	3 710 000	19.7
Type 2: 12 by 12 by 12 inches	End	4500	22.2	6980	3.4	4 267 000	7.0
	Edge	2000	25.0	4620	5.3	3 110 000	13.5
	Flat	.....	.....	4290	16.9	2 813 000	24.0
Type 3: 12 by 12 by 12 inches	End	4390	54.5	6310	15.6	3 404 900	15.7
	Edge	2600	23.1	4510	14.2	2 943 000	29.0
	Flat	2620	36.2	4310	19.2	2 977 000	29.8

TABLE 5.—Representative Tests of Tile

10 BY 12 BY 12 INCHES, TYPE 3[a]

Total load, in pounds	Load, in pounds per square inch	Berry Gage readings A	B	C	D	Differences A	B	C	D	Average of the differences	Average of the differences, corrected	Strain, in inches per inch
4 638	100	0.0306	0.0302	0.0317	0.0311	0.0000	0.0000	0.0000	0.0000	0.00000	0.00000	0 00000
23 190	500	.0310	.0304	.0330	.0324	.0004	.0002	.0013	.0013	.00080	.00076	00010
46 380	1000	.0316	.0311	.0343	.0337	.0010	.0009	.0026	.0026	.00177	.00168	00021
69 570	1500	.0324	.0319	.0351	.0349	.0018	.0017	.0034	.0038	.00267	.00254	.00032
92 760	2000	.0332	.0329	.0361	.0362	.0026	.0027	.0044	.0051	.00370	.00352	00044
115 950	2500	.0342	.0341	.0368	.0373	.0036	.0039	.0051	.0062	.00470	.00446	.00056
139 140	3000	.0351	.0353	.0376	.0383	0045	.0051	.0059	.0072	00567	.00538	00067
162 330	3500	.0361	.0366	.0382	.0393	.0055	.0064	.0065	.0082	00665	.00632	.00079
185 520	4000	.0372	.0377	.0388	.0403	.0066	.0075	.0071	.0092	.00760	.00722	00090
208 710	4500	.0384	.0386	.0397	.0413	.0078	.0084	.0080	.0102	.00860	.00817	[b] .00102
231 900	5000	.0400	.0395	.0408	.0423	.0094	.0093	.0091	.0112	.00975	.00925	.00116
255 090	5500	.0414	.0401	.0415	.0435	.0108	.0099	.0098	.0124	.01072	.01020	.00128
278 280	6000	.0430	.0409	.0426	.0448	.0124	.0107	.0109	.0137	.01192	.01132	.00142
301 470	6500	.0446	.0412	.0436	.0458	.0140	.0110	.0119	.0147	.01290	.01225	[c] .00153
407 860	[d] 8792											

12 BY 12 BY 12 INCHES, TYPE 1[e]

4 738	100	0.0290	0.0295	0.0300	0.0296	0.0000	0.0000	0.0000	0.0000	0.00000	0 00000	0.00000
23 690	500	.0293	.0302	.0318	.0313	.0003	.0007	.0018	.0017	.00112	.00106	.00013
47 380	1000	.0299	.0315	.0333	.0327	.0009	.0020	.0033	.0031	.00232	.00220	.00028
71 070	1500	.0306	.0323	.0341	.0338	.0016	.0028	.0041	.0042	.00317	.00301	.00038
94 760	2000	.0315	.0331	.0352	.0348	.0025	.0036	.0052	.0052	.00412	.00392	.00049
118 450	2500	.0325	.0340	.0363	.0355	.0035	.0045	.0063	.0059	.00505	.00480	.00060
142 140	3000	.0332	.0347	.0376	.0365	.0042	.0052	.0076	.0069	.00597	.00567	.00071
165 830	3500	.0337	.0356	.0392	.0376	.0047	.0061	.0092	.0080	.00700	.00665	.00083
189 520	4000	.0342	.0369	.0408	.0390	.0052	.0074	.0108	.0094	.00820	.00779	.00097
213 210	4500	.0344	.0380	.0421	.0403	.0054	.0085	.0121	.0107	.00917	.00872	00109
236 900	5000	.0350	.0393	.0436	.0417	.0060	.0098	.0136	.0121	.01037	.00985	.00123
260 590	5500	.0353	.0405	.0451	.0430	.0063	.0110	.0151	.0134	.01145	.01088	.00136
284 280	[f] 6000											
348 000	[g] 7350											

[a] Tested with cells vertical. Net sectional area was 46.38 square inches. Color was medium.
[b] Cracking.
[c] Shearing.
[d] Maximum load.
[e] Tested with cells vertical. Net sectional area was 47.38 square inches. Color was medium.
[f] Cracked with loud report.
[g] Maximum load.

WASHINGTON, September 18, 1918.

T
1
U55
no.109-
120

Engineering

U.S. National Bureau of Standards
Technologic papers

PLEASE DO NOT REMOVE
CARDS OR SLIPS FROM THIS POCKET

UNIVERSITY OF TORONTO LIBRARY

ENGIN STORAGE